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Fate of Semivolatile Priority Pollutants in a Wastewater Treatment Plant

Battelle Columbus Labs., OH

Prepared for

Municipal Environmental Research Lab.
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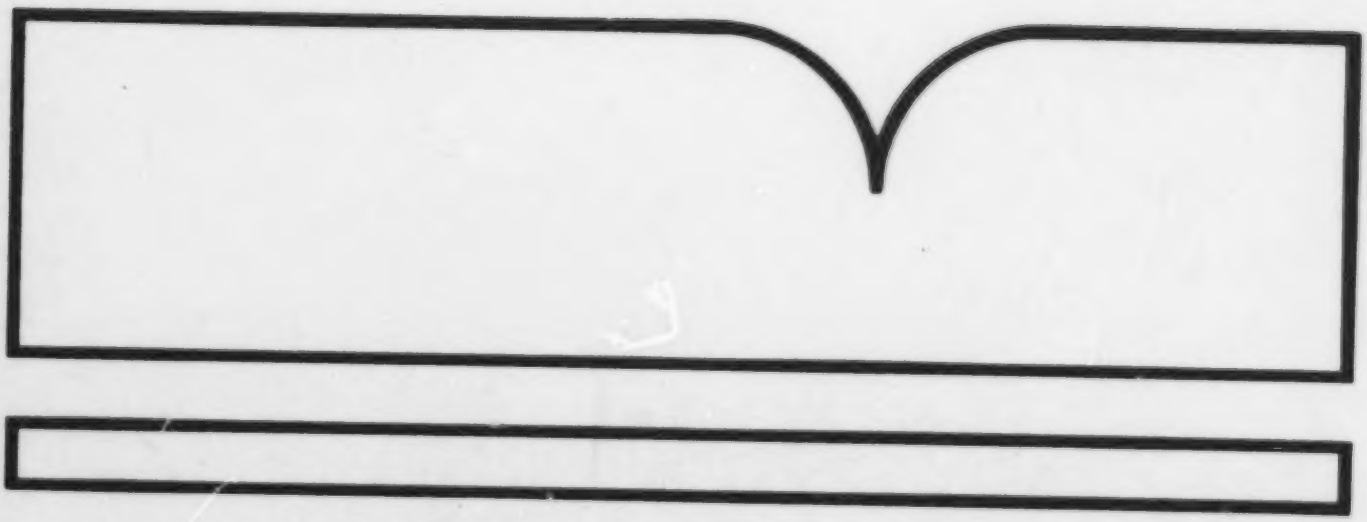

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16. ABSTRACT

This study was designed to measure the removal efficiency of a demonstration wastewater treatment plant for a series of semivolatile priority pollutants. Twenty-two representative organic priority pollutants were continuously dosed into the influent side of a dual-stream demonstration plant at the U.S. Environmental Protection Agency's Test and Evaluation Facility in Cincinnati, Ohio. These organic pollutants were administered at an average concentration of 50 ppb (W/V). Representative aqueous and sludge samples were taken at several points in the treatment sequence to study the fates of the added compounds. Wastewater samples showed the residual pollutant burden carried forward in each treatment step, and sludge samples revealed the amount of the organic pollutants retained in the sludge (or in the case of activated sludge, chemically altered by the sludge itself).

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FATE OF SEMIVOLATILE PRIORITY POLLUTANTS IN A WASTEWATER TREATMENT PLANT

by

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FOREWORD

The U.S. Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimonies to the deterioration of our natural environment. The complexity of that environment and the interplay of its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution; it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems to prevent, treat, and manage wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, to preserve and treat public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research and provides a most vital communications link between the researcher and the user community.

This report contains the results obtained from the determination of selected semivolatile priority pollutants found in a variety of wastewaters and wastewater sludges, all analyses were performed using protocol priority pollutant analytical methods.

Francis T. Mayo
Director
Municipal Environmental Research
Laboratory

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ABSTRACT

This analytical work was designed to support an evaluation of the effectiveness of a demonstration wastewater treatment plant for removing selected semivolatile priority pollutants. Twenty-two representative organic priority pollutants were continuously dosed into the influent side of a dual-stream demonstration plant at the U.S. Environmental Protection Agency Test and Evaluation Facility in Cincinnati, Ohio. These organic pollutants were added by the operators at an average concentration of 50 ppb (W/V). Representative aqueous and sludge samples were taken by the U.S. EPA staff at several points in the treatment sequence to study the fates of the added compounds. Wastewater samples showed the residual pollutant burden carried forward in each treatment step, and sludge samples revealed the amount of the organic pollutants retained in the sludge (or in the case of activated sludge, chemically altered by the sludge itself).

The second, non-spiked stream was a control sequence that provided background samples and authentic blank matrices that were spiked in the laboratory to give method recoveries.

All analyses were performed by protocol priority pollutant analytical methods utilizing WCOT (wall coated open tubular) capillary column gas chromatography with quadrupole mass spectrometric compound detection and identification (GC/MS).

This report was submitted in fulfillment of Contract No. 68-03-2552 (T2016) by Battelle Columbus Laboratories under the sponsorship of the U.S. Environmental Protection Agency. This report covers a period from April 10, 1980 to August 21, 1980, and the work was completed as of September 22, 1980.

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SECTION 1

INTRODUCTION

In support of a U.S. Environmental Protection Agency (EPA) study on municipal waste treatment, Battelle Columbus Laboratories analyzed 34 samples from a test wastewater treatment facility. The analyses were performed to determine the removal efficiency of a well-characterized municipal treatment plant for selected semivolatile priority pollutants.

A schematic diagram of the demonstration treatment plant at the Test and Evaluation Facility, Cincinnati, Ohio is shown in Figure 1. The plant offers parallel streams with well-characterized and controlled operations. One stream serves as a control and the other stream is spiked with a mixture of priority pollutants. The numbered arrows point to sampling locations at the treatment plant. Samples taken at these points are characteristic of the chemical composition of the water and sludge at various steps in the plant operation.

A simplified schematic diagram of the sampling sequence of the demonstration plant is provided in Figure 2. The sampling points are designated by an abbreviation of the treatment performed, a letter corresponding to the stream involved, and a letter, S, W, or E, indicating the nature of the sample taken at that point; S indicates sludge, W indicates wastewater, and E indicates effluent. The nominal sample size analyzed is two (2) liters for water samples and 100 grams for sludge; however, larger samples were composited and stored for possible subsequent analysis in all cases. As indicated in Figure 2, four nominally sized samples were analyzed from sample points 1, 4, 5, 6, and 7. Two of each set of four samples were subsequently spiked with reference priority pollutants and surrogate standard before analysis, and the other two samples from each set of four were spiked with only the surrogate standard. The semivolatile priority pollutant test mixture (PP), described in Table 1, was used for the reference priority pollutant spike. This mixture is a concentrated solution of 24 priority pollutants in toluene. The surrogate standard (SS) used was decafluorobiphenyl, which was chosen from the list of compounds suggested in the Federal Register, December 3, 1979.

In performing the treatment study, the same semivolatile priority pollutant test mixture was continuously added to the plant stream before sample point 8. Samples from points 8 through 14 were used to determine the fate of the injected pollutants as a result of the various treatment steps in the plant operation. Samples from points 1 through 7 were used to determine the background levels of the test priority pollutants. The replicate analyses of samples from the nonspiked treatment side were used to determine the precision and percent recovery (accuracy) of the analytical method.

The sludges and waters to be analyzed were 24-hour composite samples collected by U.S. EPA staff on three different dates at the previously designated sampling points. The samples received and the aliquots are described in Table 2.

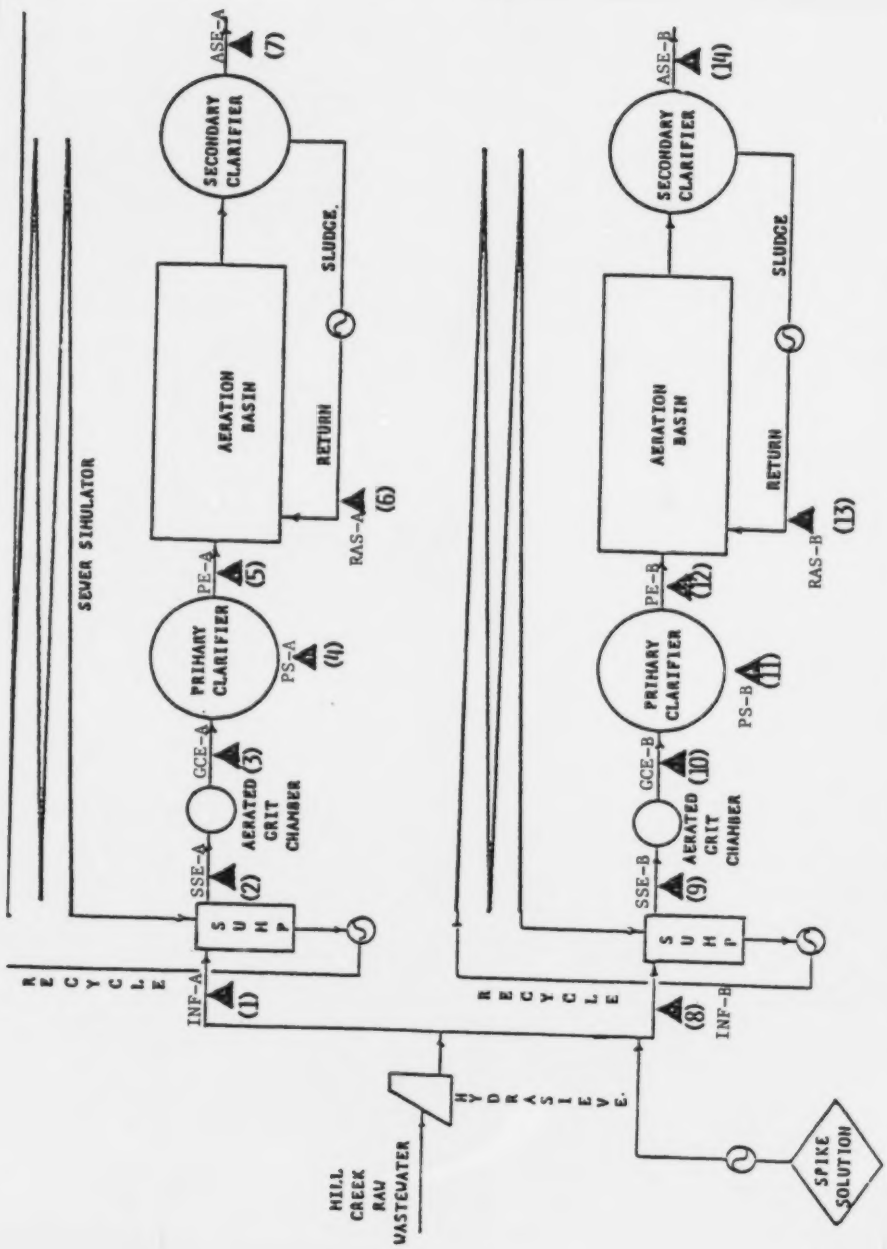
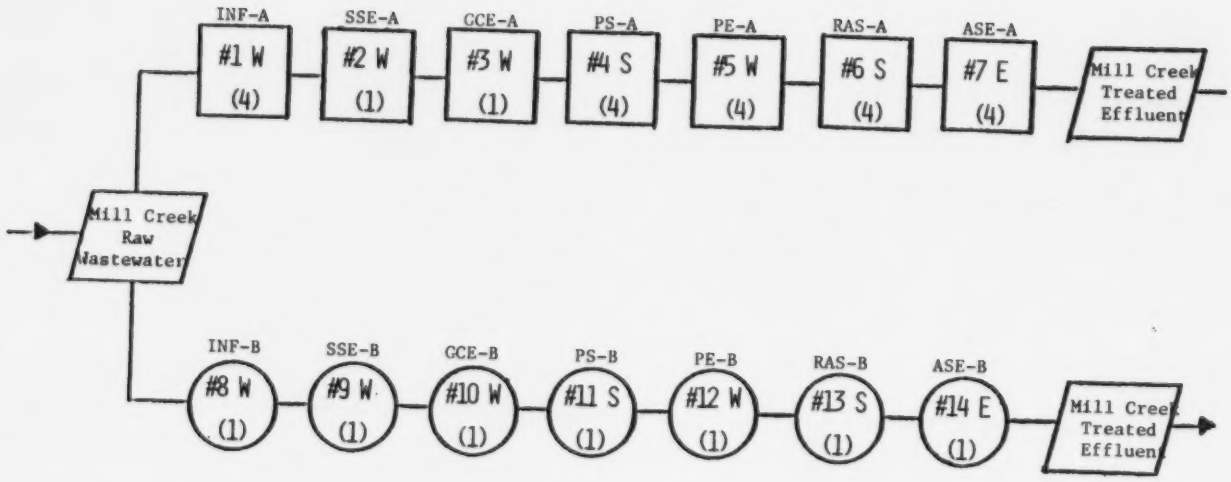


Figure 1. Schematic flow diagram of treatment demonstration plant, semivolatile priority pollutant study.



LEGEND:

E = Effluent

W = Wastewater Sample

S = Sludge Sample

( ) = Multiplicity, the number of 2 ℓ samples taken at this sampling point.

Figure 2. Schematic sampling sequence diagram of treatment demonstration plant, semivolatile priority pollutant study.

TABLE 1. COMPOSITION OF SEMIVOLATILE PRIORITY POLLUTANT TEST MIXTURE(a)

Arochlor 1254	
Toxaphene	
Phenol	Fluorene
Pentachlorophenol	Acenaphthene
2,4-Dimethylphenol	Chrysene
Naphthalene	Indeno(1,2,3-cd)pyrene
Pyrene	Di-n-octylphthalate
Benzo(a)anthracene	Butylbenzylphthalate
Di-n-butylphthalate	Dimethylphthalate
Bis-(2-ethylhexyl)phthalate	Lindane
Anthracene	Endosulfan, I and II (A and B)
Phenanthrene	Heptachlor
Fluoranthene	Diethylphthalate

(a) Arochlor 1254 and toxaphene are each present at a concentration of 4.20 mg/ml; each of the other components is present at a concentration of 1.4 mg/ml. The solvent is toluene.

TABLE 2. SAMPLES RECEIVED

Sampling Date	Date Received	Sample	Type	Volume, ℓ	Number of Aliquots Analyzed With SS	With SS&PP	Total
5/4/80	5/5/80	INF-A	water	10	2	2	4
5/4/80	5/5/80	SSE-A	water	4	1	0	1
5/4/80	5/5/80	GCE-A	water	4	1	0	1
5/4/80	5/5/80	PS-A	sludge	2	2	2	4
5/4/80	5/5/80	PE-A	water	10	2	2	4
5/4/80	5/5/80	RAS-A	sludge	2	2	2	4
5/4/80	5/5/80	ASE-A	water	10	2	2	4
5/4/80	5/5/80	INF-B	water	10	2	0	2
5/4/80	5/5/80	SSE-B	water	4	1	0	1
5/4/80	5/5/80	GCE-B	water	4	1	0	1
5/4/80	5/5/80	PS-B	sludge	2	2	0	2
5/4/80	5/5/80	PE-B	water	10	1	0	1
5/4/80	5/5/80	RAS-B	sludge	2	1	0	1
5/4/80	5/5/80	ASE-B	water	4	1	0	1
5/15/80	5/16/80	INF-A	water	10	1	2	3
5/15/80	5/19/80	PS-A	sludge	1	1	2	3
5/15/80	5/16/80	PE-A	water	10	1	0	1
5/15/80	5/16/80	RAS-A	sludge	1	1	2	3
5/15/80	5/16/80	ASE-A	water	10	1	2	3
5/15/80	5/16/80	INF-B	water	10	2	0	2
5/15/80	5/16/80	PS-B	sludge	1	2	0	2
5/15/80	5/16/80	PE-B	water	10	1	0	1
5/15/80	5/16/80	RAS-B	sludge	1	1	0	1
5/15/80	5/16/80	ASE-B	water	4	1	0	1
5/28/80	5/29/80	INF-A	water	10	1	2	3
5/28/80	5/29/80	PS-A	sludge	1	1	2	3
5/28/80	5/29/80	PE-A	water	10	1	0	1
5/28/80	5/29/80	RAS-A	sludge	1	1	2	3
5/28/80	5/29/80	ASE-A	water	10	1	2	3
5/28/80	5/29/80	INF-B	water	10	2	0	2
5/28/80	5/29/80	PS-B	sludge	1	2	0	2
5/28/80	5/29/80	PE-A	water	10	1	0	1
5/28/80	5/29/80	RAS-B	sludge	1	1	0	1
5/28/80	5/29/80	ASE-B	water	4	1	0	1
						Grand Total	71

NOTE: In all cases, the samples from stream B were significantly different from the corresponding samples from stream A. The differences included color, turbidity, suspended solids, odor, and in some cases, foaming during extraction and separation by centrifugation.

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SECTION 2

CONCLUSIONS

The procedures used in and the results obtained from the analysis of three sets of samples collected from a demonstration wastewater treatment facility are presented in this report. Each set of samples consisted of wastewater and sludge samples from two parallel treatment streams, one being a control and one being spiked with a solution containing selected semivolatile priority pollutants.

Recovery of test compounds from wastewater was generally good. The recoveries from sludge were consistently lower and showed much more variability. Recoveries from primary sludge were lower than from returned activated sludge. These data indicate that although the methods used represent best current methodology, further optimization is needed to improve recovery and precision.

Phenol and napthalene were consistently present in high concentrations in the control stream. This unusually high background sometimes made it impossible to quantify these two compounds accurately. This background was a continually recurring problem with phenol. Lindane, several PAH compounds, and several phthalates were also occasionally present in varying concentrations in the background samples.

Several compounds were found to exhibit erratic behavior. The special problem posed by phenol and napthalene has been mentioned. Additionally, 2,4-dimethylphenol, dimethylphthalate and diethylphthalate appeared to give much more varied results than other compounds in duplicate analyses or analyses of similar samples. All compounds showed at least one incidence of apparently inconsistent results.

Toxaphene and Arochlor 1254, being mixtures of many compounds and isomers, show lower mass spectral sensitivity than do single component materials. Although present at three times the levels of the other analytes, far greater concentrations would be required to achieve chromatographic peaks of the major constituents which are equal to those of the single component species. Moreover, these mixtures elute in a very complex region of the chromatogram, thereby increasing the difficulty in selecting the appropriate peak from those of other unrelated compounds having the same mass. Further complicating detection and quantification of these multicomponent materials is the fact that the mass spectra are very complex and do not contain predominant, or otherwise desirable, quantification ions. The higher detection limit and reduced reproducibility observed for this series of compounds are a result of these complications.

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SECTION 3

EXPERIMENTAL STUDIES

The analytical approach used to characterize the samples consisted of extraction of the pollutants from each sample, concentration and purification of the extracts, and analysis by glass capillary gas chromatography-mass spectrometry (GC/MS). Due to the intrinsic matrix differences between wastewater and sludge, two different procedures were used to prepare the samples for GC/MS analysis. A flow chart of the analytical sequence is given in Figure 3.

EPA Method 625 (Federal Register, December 3, 1979, "Guidelines Establishing Test Procedures for the Analysis of Pollutants; Proposed Regulations," pp. 69540-69552) was used for GC/MS analysis of extractable, semivolatile priority pollutants in the wastewater samples. The extractions were performed using all-glass continuous extractors, suggested in the Federal Register as an alternative to separatory funnels.

The GC/MS analyses were performed using a glass capillary column system, suggested in the Federal Register as an alternative to a packed column system. Unlike the packed column system, which requires that the base-neutral and acid fractions be analyzed separately using two different columns, the capillary GC/MS system is capable of analyzing both fractions at once. Consequently the base-neutral and acid fractions were combined prior to analysis. This combination of extracts is the only deviation from Method 625 that was adopted for this study. The surrogate standard (SS) and, where appropriate, the priority pollutant (PP) solution, were spiked into the wastewater at the levels indicated in Table 3. The solutions were added and the sample stirred for 45 minutes prior to starting the extraction.

TABLE 3. SPIKING LEVELS OF SURROGATE STANDARD AND PRIORITY POLLUTANT SOLUTIONS (ppb)

Item	SS	PP
Wastewater	100	140
Sludge	1000	1400

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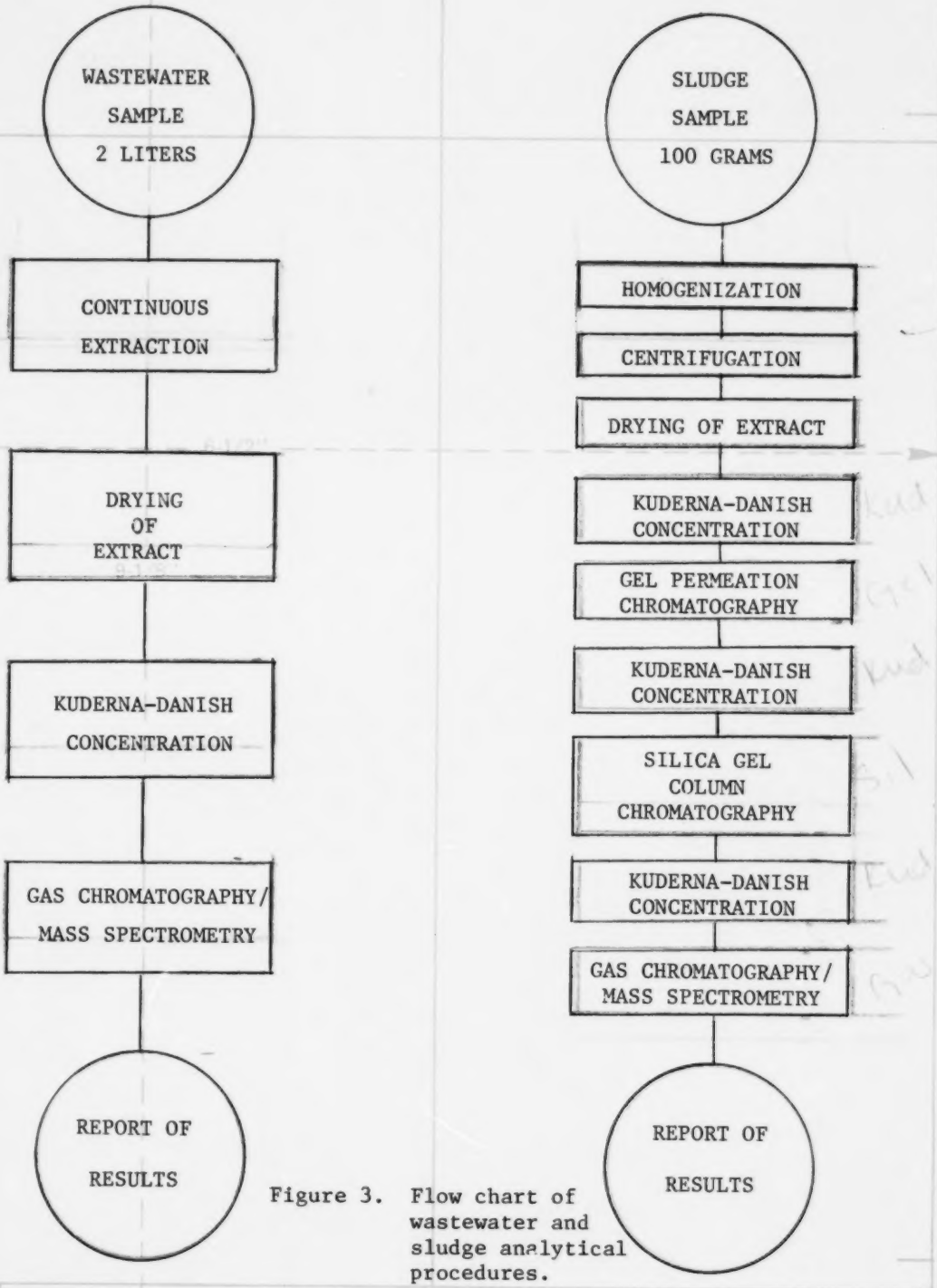

Figure 3. Flow chart of wastewater and sludge analytical procedures.

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A detailed description of the method used for sludge preparation is found in Appendix A. Only a brief outline is presented here. Sludge samples were prepared by spiking 100 g of each sample at the level indicated in Table 3, homogenizing with a Tekmar "Tissumizer", and storing the homogenate for 24 hours at 4°C. The samples were then acidified to pH 2, extracted with methylene chloride using a Tekmar "Tissumizer", and centrifuged. Each methylene chloride extract was separated from the aqueous sample using a syringe and was concentrated using a Kuderna-Danish evaporator. Gel permeation chromatography (GPC) and silica gel column chromatography were used to remove interfering species. The fractionated extracts were concentrated using a stream of dry nitrogen and analyzed by GC/MS. Residue weights on all samples analyzed by GC/MS were obtained by weighing residues from sample aliquots on a microbalance. For sludge samples this operation was also done at the pre-cleanup stage.

The GC/MS system used for quantitative analyses consisted of a Finnigan Model 9500 biomedical-type gas chromatograph coupled with a Finnigan Model 3200 mass spectrometer via a single-piece, fused silica capillary transfer line (0.2 mm ID) which was deactivated with Carbowax 20 M. Data collection and processing were handled by a Finnigan "Incos Data System" which utilizes a "Data General Nova 3" computer with 32 K of memory. The GC/MS operating conditions for these analyses are given in Table 4. The pertinent GC/MS parameters for compound identification and quantification are given in Table 5. Compound identification was based on the presence of the three ions labeled P_1, P_2, P_3, as well as the relative retention time. After identification, quantification was based on a single ion, labeled Q_1. The area of Q_1 was ratioed to that of the internal standard, d_{10}-anthracene. The d_{10}-anthracene internal standard was added to all sample extracts at the 10 µg per milliliter level at the time of analysis.

TABLE 4. GC/MS OPERATING CONDITIONS

Item	Description
Chromatography:	
Column	SE-52
Length	30 m
Carrier	He
Velocity	30 cm/sec at 25°C
Injection Volume	2 µl
Injection Mode	Splitless
Temperature Program:	
Column Initial	50°C
Hold Time	3 min.
First Program Rate	20°/min. to 100°C
Hold Time	30 sec.
Second Program Rate	4°/min. to 280°C
Final Hold Time	10 min.
Transfer Line	270°C
Mass Spectrometer	
Ionization	EI at 70 eV
Emission Current	0.10 µA
Pre-amplifier	10^{-7} amp/volt
Electron Multiplier	10^{4} gain at 1.4 KV
Scan Range	40-450

TABLE 5. RELATIVE RETENTION TIMES AND IONS USED FOR GC/MS DATA SEARCHING AND QUANTIFICATION OF DESIGNATED PRIORITY POLLUTANTS

Pollutant Species	M. W.	Relative Retention Time(a)	Mass Spectral Ions			
			Data Search			Quantification
			P_1	P_2	P_3	Q_1
Phenol	94	0.259	94	65	66	94
Decafluorobiphenyl	334	0.350	334	265	167	334
2,4-Dimethylphenol	122	0.374	122	107	121	122
Naphthalene	128	0.414	128	129	127	128
Dimethylphthalate	194	0.674	163	194	164	163
Acenaphthene	154	0.707	154	153	152	154
Fluorene	166	0.805	166	165	167	166
Diethylphthalate	222	0.814	149	177	150	149
Lindane	290	0.980	183	181	109	183
Phenanthrene	178	0.995	178	179	176	178
d_{10}-Anthracene	188	1.000	188	189	186	188
Anthracene	178	1.006	178	179	176	178
Heptachlor	373	1.107	100	272	274	100
Di-n-butylphthalate	278	1.156	149	150	104	149
Fluoranthene	202	1.247	202	101	100	202
Pyrene	202	1.291	202	101	100	202
Toxaphene		1.299	231	233	235	235
Butylbenzylphthalate	298	1.472	149	91		149
Benzo(a)anthracene	228	1.551	228	229	226	228
Chrysene	228	1.560	228	229	226	228
Bis(2-ethylhexyl)phthalate	390	1.608	149	167	279	149
Di-n-octylphthalate	390	1.736	149	167	279	149
Arochlor 1254						
Tetrachlorobiphenyl	292	1.245	220	290	292	292
Pentachlorobiphenyl	326	1.391	254	326	328	326
Hexachlorobiphenyl	360	1.480	290	360	362	360

(a) Relative to that of d_{10}-Anthracene.

SECTION 4

RESULTS

The results of the GC/MS analyses of wastewater and sludge are given in Tables B1 to B6 (Appendix B). Summaries of Control Stream A and Spiked Stream B are given in Tables B7 and B8. Samples are identified in these tables according to the sample naming system used at the treatment facility, as indicated in Table 2. To this sample designation is added SS (surrogate standard) and, where appropriate, PP (priority pollutant) to indicate which type of sample spiking was employed. Roman numerals are used to differentiate between replicates.

These data indicate a consistently high background level of phenol and naphthalene in the Mill Creek influent. Also present as background in varying concentrations were several PAH compounds, as well as several phthalates. Unexpectedly, lindane was found in the unspiked influent of May 15. For each day's set of samples, the final effluent from experimental stream B shows lower levels of pollutants than were present in the influent. The primary sludge samples consistently contained higher levels of the compounds of interest than the returned activated sludge samples.

Tables B9 and B10 give the residue weights for all samples. For sludge samples, residues were determined before and after clean-up procedures were performed.

Chromatograms representative of those obtained during analysis are given in Figures B1 to B5. A reconstructed ion chromatogram (RIC) of a standard used in the study is shown in Figure B1. This standard represents the stated limit of detection (20 ng for all compounds, with the exception of the Arochlor 1254 and toxaphene at 60 ng each). As shown, the system in use easily meets the required detection limit. Successful separation of isomers, phenanthrene and anthracene, and benzo(a)anthracene and chrysene, as well as the consistently sharp peaks throughout the analysis should be noted.

The RICs of an influent and a final effluent from spiked stream B of the treatment system are shown in Figures B2 and B3. The decreased complexity of the chromatogram of the effluent reflects a decrease in pollutant levels from those present in the influent, a trend confirmed by quantification of the designated priority pollutants.

Chromatograms representative of sludge samples, given in Figures B4 and B5, are from equivalent sampling points in the control and spiked streams. In

contrast to the wastewater influent and effluent chromatograms, the extreme complexity of these sludge samples make it difficult to differentiate between the control and spiked samples. The sample complexity also has a significant effect on the quantification of the designated priority pollutant analytes.

SECTION 5

CALIBRATION AND QUALITY ASSURANCE

The purpose of quality assurance is to establish the precision and accuracy of the results relative to those reference or standard materials available so that levels of confidence can be assigned to reported data. During this study several measures of quality assurance and quality control were applied.

Blanks were analyzed with each set of water and sludge samples to determine background contributions and to test for potential contamination. No samples of distilled water or other solvents used during sampling and standards preparation were available. The data given in Table C1 (Appendix C) are from blanks created in the laboratory using distilled water and solvents used for extraction.

The standards used for identification and calibration were prepared by Supelco Inc. and were supplied by EPA. The compounds reported to be present in the solution are listed in Table 1. During tuning and calibration checks, it was found that pentachlorophenol, indeno-(1,2,3-cd)pyrene, and Endosulfan I and II were not present or were below the limit of detection. Since these compounds were not present in the standard in measurable amounts, they were deleted from the list of designated analytes.

Standards were analyzed by GC/MS in order to determine relative response factors for each compound over a wide range of concentrations. These calibration analyses were performed and evaluated before sample analysis was begun. Equation 1 defines the formula used to calculate relative response factors.

$$RF = \frac{A_s \, C_{is}}{A_{is} \, C_s} \qquad (1)$$

where A_s = the integrated area of the characteristic ion for the pollutant standard

A_{is} = the integrated area of the characteristic ion for the internal standard

C_{is} = the amount (µg) of the internal standard

C_s = the amount (µg) of the pollutant standard

Standards were analyzed in triplicate at the 5 levels shown in Table 6. The average of three determinations of each level, as well as the mean response factors over the entire range are shown in Table C2.

Prior to initiating sample analyses, the recovery of the target compounds was verified by spiking distilled water with the standard solutions. As can be seen in Table C3 the mean recoveries varied from 50% to 122%, and averaged 76%.

The experimental design included the analysis of duplicates and spikes. Samples from control stream A were split into several portions and spiked as shown in Figure 4. A detailed characterization of Mill Creek and the recovery of the selected priority pollutants from this matrix were obtained from the data. All recoveries were calculated according to Equation 2. A detailed presentation of recovery data is given in Tables C4 and C5. For wastewater the average recoveries range from 20% to 110%, and averaged 71%. These data can be compared to the recoveries obtained from initial recovery studies using distilled water where the recoveries varied from 50% to 122%.

$$\% \text{ Recovery} = \frac{M - P}{S} \times 100 \qquad (2)$$

where M = the value measured in the spiked sample extract

P = the amount originally present as measured in the unspiked sample extract

S = the amount spiked into the sample

For sludge the recoveries range from 4.1% to 67%, averaging 36%. As expected, due to the extremely complex nature of the sample, recoveries for sludge were in all cases lower and more variable than for wastewater samples.

Observed recoveries according to sample type shown in Tables C6 and C7, indicate good agreement between similar sample types, but again emphasize the disparity between sludge and water.

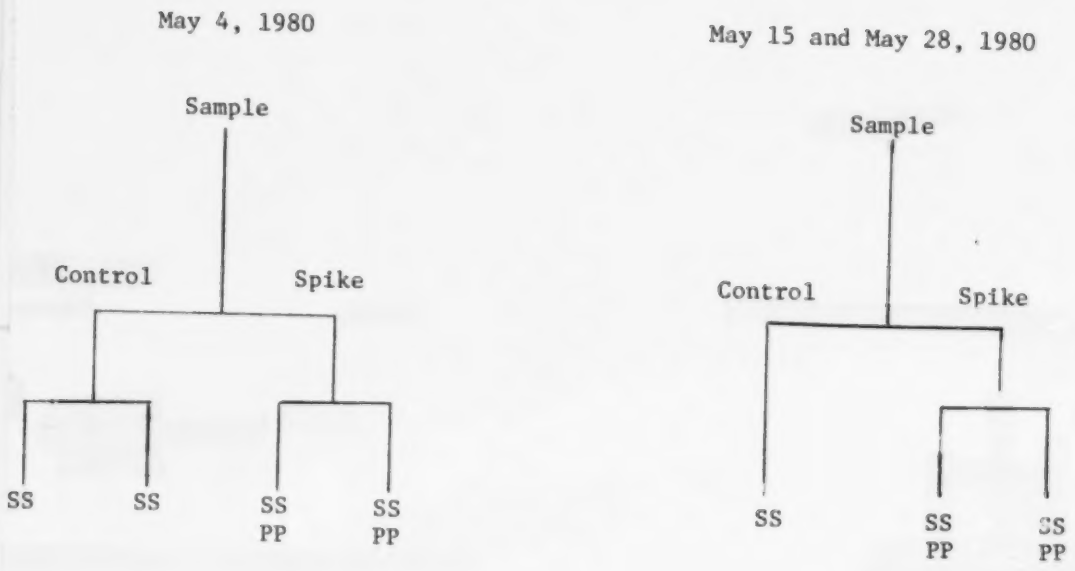
A standard was analyzed after every fourth sample and quantified using the previously determined response factors. The results of these analyses are given in Table C8. The mean of all the determinations along with a measure of variance, relative standard deviation [RSD = $(s/\bar{x}) \times 100$], and a measure of accuracy, mean deviation [MD = $\frac{(\mu - \bar{x})}{\mu} \times 100$], are given in Table C9. The date provide an indication of the precision and accuracy of the GC/MS measurements obtained throughout the course of GC/MS analyses. This continual re-evaluation of standard solutions and the consistency of quantification indicate that little compound degradation in the standard solutions occurred under sample storage conditions.

Each day prior to sample analysis, the mass spectrometer tuning was checked. Perfluorotributylamine (FC-43) was introduced into the instrument, and the peak shape, resolution, and high mass-low mass balance were adjusted. After obtaining mass spectra which satisfied rigid ion ratio requirements,

decafluorotriphenylphosphine (DFTPP) was injected into the instrument. The peak height and retention time provided monitors of instrument sensitivity and possible degradation of the chromatographic system. The ion ratios for DFTPP listed in the Federal Register, December 3, 1979, were met before analyses were begun. Meeting strict ion ratio requirements for both FC-43 and DFTPP ensured that the mass spectrometer was tuned the same way each day. This step was particularly important considering the broad range of masses used for identification and quantification.

TABLE 6. CONCENTRATION OF PRIORITY POLLUTANTS AND INTERNAL STANDARDS IN CALIBRATION STANDARDS (µg/ml)

	Std. A	Std. B	Std. C	Std. D	Std. E
Priority Pollutants	2.5	10	50	100	250
Surrogate Standard	2.5	10	50	100	250
d_{10}-Anthracene	10	10	10	10	10



where SS = Surrogate Standard Spike

PP = Priority Pollutant Spike

Figure 4. Sequence for division of control stream samples.

APPENDIX A

METHOD FOR SEMIVOLATILE ORGANIC COMPONENTS IN SLUDGE

Scope and Application

This method covers the determination of and is applicable to semivolatile organic priority pollutants in raw and digested municipal sludge.

Summary

The procedure involves the use of repetitive solvent extraction to recover components from the sludge matrix efficiently. Gel permeation chromatography and silica gel chromatography are used as cleanup procedures to eliminate interferences. The acidic (phenolic) and neutral components are determined by GC/MS analysis using high-resolution glass capillary columns and selected ion searches. The overall scheme is shown in Figure A-1.

Apparatus and Reagents

For sample extraction:

- Tekmar "Tissumizer"
- Centrifuge, refrigerated, capable of handling 200-ml tubes
- Distilled-in-glass grade methylene chloride
- Centrifuge tubes - 200-ml, with Teflon-lined screw caps
- Separatory funnel - 250-ml, with Teflon stopcocks
- Kuderna-Danish (K-D) glassware
  - Synder column - 3 bulb, macro
  - Evaporating flask, 500-ml
  - Evaporator tubes - 10-ml and 25-ml, graduated, with spring attachment
  - Glass stoppers for evaporator tubes
- Reagents
  - Sodium sulfate, anydrous - conditioned at 450°C
  - Sulfuric acid, 50%
- Syringe - 50-ml with 8-inch 15-gauge square-tipped needle
- Microbalance
- Aluminum foil pans - 25-mm

For removal of interferences:

- Reagents
  - Sodium sulfate, anhydrous - conditioned at 450°C

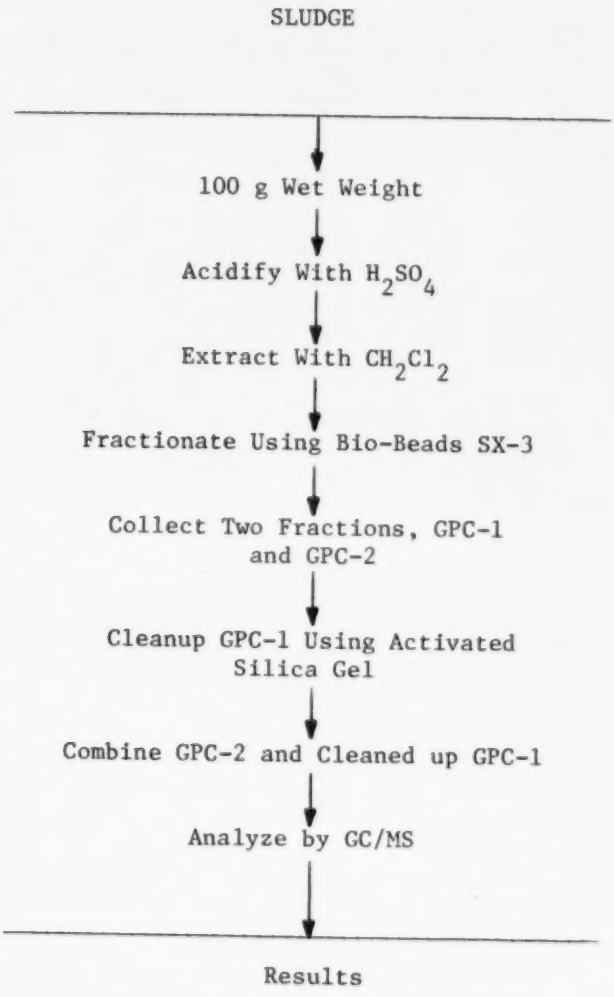


Figure A-1. Scheme for determination of semivolatile priority pollutants in sludge.

- Glassware
  - Separatory funnels - 250-ml with Teflon stopcocks
  - Kuderna-Danish glassware
  - Centrifuge tubes - 200-ml with Teflon-lined screw cap
  - Chromatography column - 400-mm x 9-mm I.D. Lab-Crest column with 100-ml reservoir, sintered glass frit (medium porosity), Teflon stopcock, and ground glass joints
- Solvents, distilled-in-glass grade
  - Methylene chloride
  - Petroleum ether, b.p. 30-60°C
  - Acetone
- Silica gel - 100/200-mesh Davison, grade 923, activated for 16 hours at 150°C
- Standard solution
  GPC calibration solution for acid/neutrals - methylene chloride containing corn oil, di-n-octylphthalate, 2,4-dinitro-6-methyl phenol, pentachlorobiphenyl, and sulfur
- Gel permeation chromatography system
  - Chromatographic column - 500-mm x 25-mm I.D. glass column fitted for upward flow operation
  - Bio-Beads S-X3 - 50 g per column
  - GPC Auto Prep 1001, Analytical Biochemistry Laboratories Inc., with a flow rate of 5 ml/min
  - Altex "Model 150" ultraviolet detector - 254-nm
- Centrifuge, refrigerated, capable of handling 200-ml tubes
- Syringe - 10-µl

Sample Extraction - Acid/Neutral Fraction

- Place 100 g of sludge in a 200-ml centrifuge tube, spike with appropriate solutions, homogenize, and store at 4°C for 24 hours.
- At room temperature, acidify the spiked sample to pH 2 with H_2SO_4.
- Add 75 ml methylene chloride to the centrifuge tube and homogenize for one minute with a "Tissumizer".
- Cap the centrifuge tubes tightly with Teflon-lined screw caps and centrifuge at 15°C and 2500 rpm for 30 minutes to achieve good phase separation. Remove the methylene chloride layer with a 50-ml syringe.
- Repeat extraction procedure two more times.
- Dry the extract by using a sodium sulfate chromatographic column, as described in the Federal Register, December 3, 1979.
- Collect the extract in a 500-ml Kuderna-Danish apparatus and concentrate to 10 ml. Obtain a residue weight on 100 µl of the 10-ml concentrate, and calculate the total amount of material in the sample. The residue weight is determined by placing the 100 µl on a tared aluminum foil pan, allowing the solvent to evaporate, and reweighing the pan using a microbalance. Concentrate the extract further, as necessary, to adjust the final total volume to 1.0 ml for every 200 mg of material in the sample. Centrifuge the concentrate to remove traces of particulate material.

Removal of Interferences

The cleanup procedures described in this section are designed to remove the major interfering classes of compounds found in sludge extracts, namely triglycerides, fatty acids, and long-chain hydrocarbons. Gel permeation chromatography is used to remove triglycerides and to obtain and intermediate-sized molecule fraction containing the remaining neutral compounds of interest. Siliga gel chromatography is used to remove fatty acids and saturated hydrocarbons from the higher alkyl phthalates. A flow diagram of the cleanup procedures is shown in Figure A-2.

- Prepare a 500-mm x 25-mm I.D. gel permeation chromatography (GPC) column by slurry packing using 50 g of Bio Beads S-X3 that have been swelled in methylene chloride for at least 4 hours. Prior to initial use, rinse the packed column with methylene chloride at 1 ml/min for 16 hours to remove any traces of contaminants. Calibrate the system by injecting a GPC calibration solution for acid/neutrals, eluting with methylene chloride at 5 ml/min and observing the resultant UV detector trace. A representative chromatogram is shown in Figure A-3. The column may be used indefinitely as long as no darkening or pressure increases occur and a column efficiency of at least 500 theoretical plates is achieved. The pressure should not be permitted to exceed 100 psi. Recalibrate the system daily.
- Inject the acid/neutral concentrate onto the GPC column and elute with methylene chloride at 5 ml/min. Discard the first fraction that elutes up to a retention time represented by the minimum between the corn oil peak and the di-n-octylphthalate peak in the calibration run, usually about 25 minutes. Collect as Fraction GPC-1 the next fraction eluting up to a retention time represented by the minimum between the di-n-octylphthalate peak and the 2,4-dinitro-6-methyl-phenol peak in the calibration run, usually about 32 minutes. Collect as Fraction GPC-2 the remaining eluate the elutes up to a retention time represented by the minimum between the pentachlorophenol peak and the sulfur peak in the calibration run, usually about 45 minutes.
- Prepare a 400-mm x 9-mm I.D. silica gel chromatography column by packing using a slurry of 20 g of activated silica gel suspended in 25% acetone/75% methylene chloride. Wash the column, using gravity flow, with 50 ml of 25% acetone/75% methylene chloride to remove any traces of impurities and then wash it with 50 ml of petroleum ether to remove the polar solvent. Care should be taken to avoid any bubbles of air or solvent vapor in the column. Solvent flow should be continued only until the solvent level is within 1 mm of the top of the silica gel.
- Concentrate Fraction GPC-1 to 2 ml using a Kuderna-Danish apparatus and add 2 ml of petroleum ether.
- Apply the Fraction GPC-1 concentrate to the freshly prepared silica gel column and open the stopcock to permit flow until the liquid level is within 1 mm of the top of the silica gel. In a similar fashion rinse the sample onto the column completely with two 1-ml portions of 50% methylene chloride in petroleum ether.
- Elute the column with 50 ml of 50% methylene chloride in petroleum ether to remove interfering nonpolar and slightly polar components. Discard this eluate.

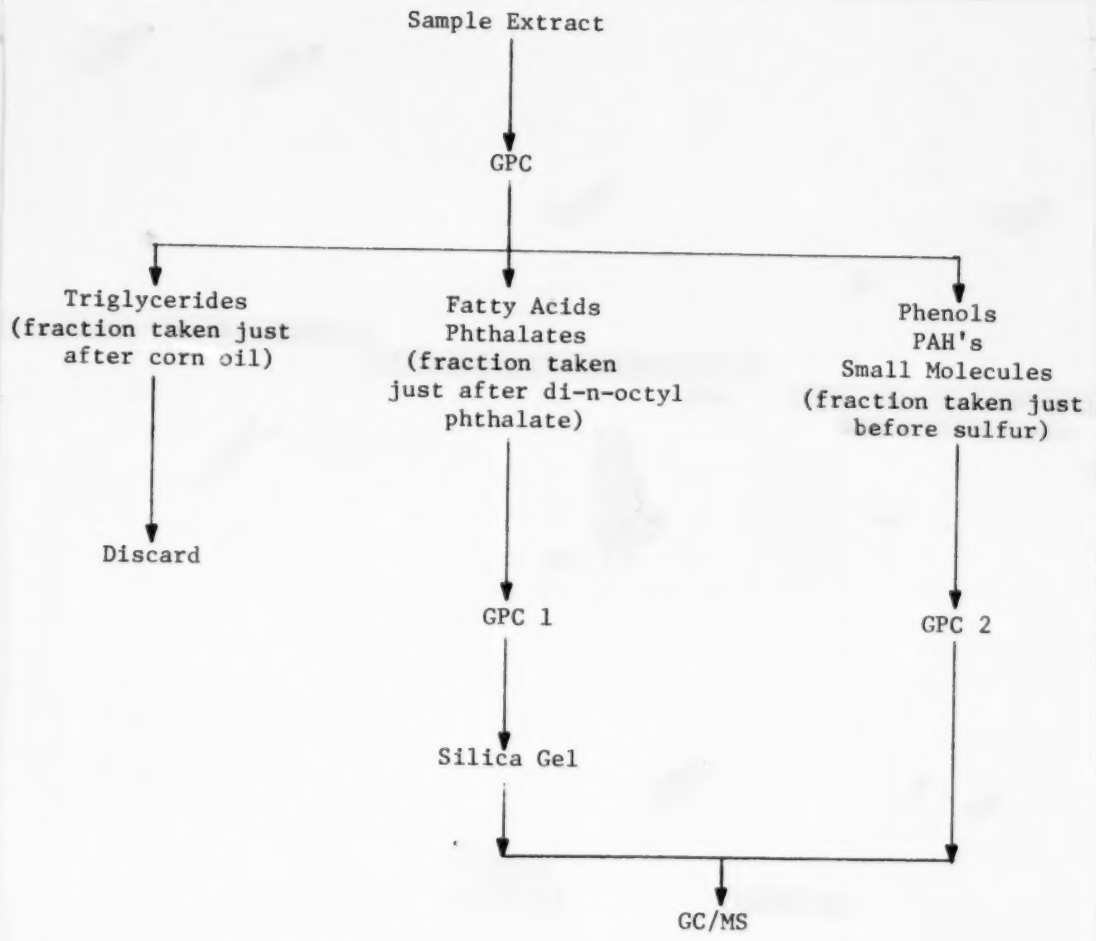
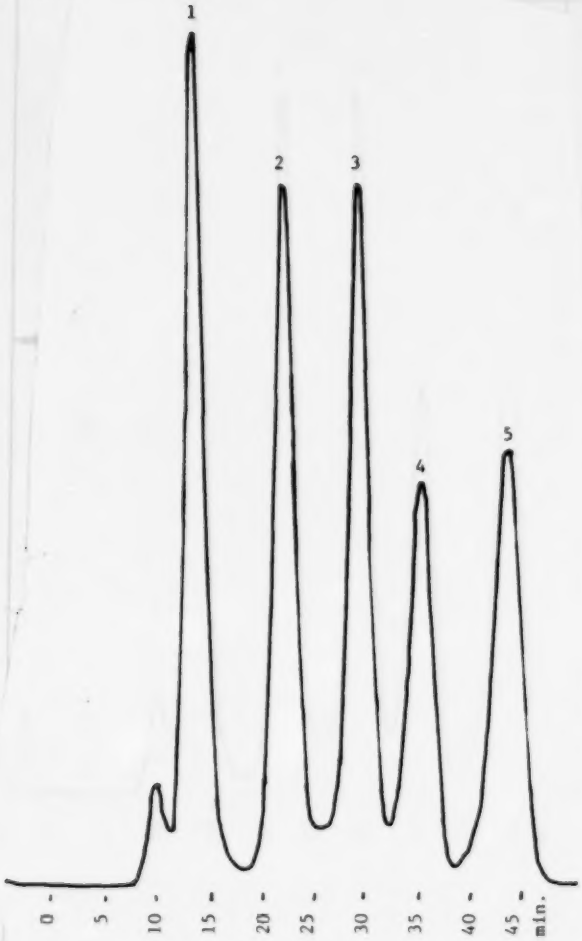


Figure A-2. Scheme for removal of interferences from sludge.



Components of GPC Standard

1. corn oil - 1 mg/ml
2. di-n-octylphthalate, 0.2 mg/ml
3. 2,4-dinitro-6-methylphenol, 0.1 mg/ml
4. pentachlorophenol, 0.5 mg/ml
5. sulfur, 0.2 mg/ml

GPC Conditions

Column S-X3, Length 500 mm, I.D. 25 mm

Packing 200/400 S-X3 Bio Beads; Chart Speed 12 cm/hr

Solvent CH_2Cl_2; Temperature 27°C

Flow Rate, 5 ml/min; Pressure 7.5 lb

Detector, Altex #150 UV; Injection Volume, 5 ml

Figure A-3. Chromatogram of GPC standard.

- Elute the adsorbent with 50 ml of 25% acetone/75% methylene chloride and collect the eluate as the phthalate fraction. The highly polar components (e.g. fatty acids) remain on the silica gel which is discarded.
- Combine the phthalate fraction with Fraction GPC-2 and concentrate to 1 ml using a Kuderna-Danish apparatus. This is the final fraction used for GC/MS analysis.

APPENDIX B

GC/MS RESIDUE DATA

TABLE B-1. QUANTIFICATION OF PRIORITY POLLUTANTS IN WASTEWATER SAMPLED 5/4/80 (ppb)

	INF-A SSI	INF-A SSII	INF-A SSII	INF-A SSPPIII	INF-A SSPPIV	SSE-A SSI	GCE-A SSI	PE-A SSI	PE-A SSII	PE-A SSPPIII	PE-A SSPPIII	PE-A SSPPIV	ASE-A SSI	ASE-A SSII	ASE-A SSPPIII	ASE-A SSPPIV
Pesticides																
Arochlor 1254																
Tetrachlorobiphenyl[a]	<3	<3	<3	220	330	<3	<3	<3	<3	310	280	310	<3	<3	280	420
Pentachlorobiphenyl[a]	<3	<3	<3	250	340	<3	<3	<3	<3	300	300	300	<3	<3	280	300
Hexachlorobiphenyl[a]	<3	<3	<3	240	310	<3	<3	<3	<3	270	250	250	<3	<3	220	250
Heptachlor	<1	<1	<1	110	190	<1	<1	<1	<1	170	180	200	<1	<1	250	280
Lindane	<1	<1	<1	80	140	<1	<1	<1	<1	160	290	180	<1	<1	120	130
Toxaphene[a]	<3	<3	<3	270	490	<3	<3	<3	<3	420	300	310	<3	<3	330	360
Phenols																
2,4-Dimethylphenol	<1	2.7	<1	75	130	<1	<1	<1	<1	130	130	140	<1	<1	39	43
Phenol	160[b,c]	240[b,c]	240[b,c]	150[b,c]	300[b,c]	490[b,c]	420[b,c]	350[b,c]	400[b,c]	450(b)	310(b)	410(b)	9	9	130	160
Phthalates																
Bis(2-ethylhexyl)phthalate	21	25	20	100	160	20	18	10	12	170	160	170	3.3	4.1	210	240
Butylbenzylphthalate	3.7	5	4.4	110	170	6	1.7	2.6	2.1	170	160	170	<1	<1	200	230
Diethylphthalate	1.3	2.2	2.2	55	100	2.1	1.5	2.6	1.8	95	90	110	<1	0.6	80	100
Dimethylphthalate	<1	<1	<1	32	70	<1	<1	<1	<1	50	48	80	<1	<1	4.6	6.5
Di-n-butylphthalate	2.4	3.6	3.9	70	120	4.5	2.7	3.2	3.7	160	120	160	1.0	1.0	200	210
Di-n-octylphthalate	2.0	<1	<1	110	170	<1	<1	<1	<1	190	180	190	<1	<1	230	260
PAHs																
Acenaphthene	<1	<1	1.1	65	120	1.1	1.8	1.0	1.2	120	110	120	<1	<1	140	150
Anthracene	<1	<1	<1	65	100	<1	<1	<1	<1	110	100	120	<1	<1	120	130
Benzo(a)anthracene	1.5	<1	<1	100	140	<1	<1	<1	<1	140	130	130	<1	<1	140	160
Chrysene	1.3	<1	<1	85	120	<1	<1	<1	<1	110	240	110	<1	<1	110	130
Decafluorobiphenyl	30	38	36	29	45	45	20	34	39	50	45	49	55	30	50	55
Fluoranthene	<1	<1	<1	70	120	<1	<1	<1	<1	120	110	115	<1	<1	130	150
Fluorene	<1	<1	<1	75	120	2.6	2.0	3.1	1.5	120	120	130	<1	<1	150	150
Naphthalene	29	39	39	55	100	80	85	60	60	170	110	160	1.8	2.6	140	160
Phenanthrene	2.6	2.6	2.7	70	110	4.0	3.7	2.1	2.5	120	110	120	<1	<1	140	160
Pyrene	<1	<1	<1	70	110	<1	<1	<1	<1	120	100	110	<1	<1	130	140

Footnotes appear on the following page.

TABLE B-1. (Continued)

	INF-B SSI	INF-B SSI	INF-B SSII	SSE-B SSI	GCE-B SSI	PE-B SSI	ASE-B SSI	Water Blank SSI
Pesticides								
Arochlor 1254								
Tetrachlorobiphenyl[a]	10	11	10	24	23	12	<3	<3
Pentachlorobiphenyl[a]	12	13	11	22	26	14	<3	<3
Hexachlorobiphenyl[a]	12	12	11	18	28	14	<3	<3
Heptachlor	22	20	20	19	28	18	4.7	<1
Lindane	15	5	6	4.2		4.8	4.4	<1
Toxaphene[a]	7	<3	28	<3	5.5	<3	<3	<3
Phenols								
2,4-Dimethylphenol	50	55	60	55	60	55	<1	<1
Phenol		240+[b]	260+[b]	260+[b]	190[b]	180+[b]	1.6	<1
Phthalates								
Bis(2-ethylhexyl)phthalate	48	45	43	50	85	60	7	<1
Butylbenzylphthalate	8	8	7.5	8.5	8.5	7	<1	<1
Diethylphthalate	8.5	8.5	9	8.5	8.5	12	<1	<1
Dimethylphthalate	1.2	2.0	2.6	2.1	2.2	1.8	<1	<1
Di-n-butylphthalate	9.5	10	10	10	15	16	<1	<1
Di-n-octylphthalate	28	29	26	38	60	40	<1	<1
PAHs								
Acenaphthene	35	36	36	41	45	37	<1	<1
Anthracene	23	23	22	22	31	18	<1	<1
Benzo(a)anthracene	20	23	18	32	40	24	<1	<1
Chrysene	17	16	15	23	33	15	<1	<1
Decafluorobiphenyl	24	26	28	36	45	48	39	43
Fluoranthene	21	17	16	24	33	20	<1	<1
Fluorene	34	35	34	39	45	37	<1	<1
Naphthalene	46	45	49	41	70	45	<1	<1
Phenanthrene	31	29	30	42	46	33	<1	<1
Pyrene	19	16	16	23	32	19	<1	<1

(a) The single most intense isomer was used to calculate the total amount of Arochlor 1254 and toxaphene.

(b) Data system saturated. Actual value is higher than this.

(c) Apparent outlier or incongrous data.

TABLE B-2. QUANTIFICATION OF PRIORITY POLLUTANTS IN SLUDGE SAMPLED 5/4/80 (ppb)

	Sludge Blank	Sludge Blank	PS-A SSI	PS-A SSII	PS-A SSPPIII	PS-A SSPPIII	PS-A SSPPIV	PS-B SSII	RAS-A SSI	RAS-A SSII	RAS-A SSPPIII	RAS-A SSPPIII	RAS-A SSPPIV	RAS-B SSI
Pesticides														
Arochlor 1254														
Tetrachlorobiphenyl[a]	<60	<60	<60	<60	<60	<60	2200 [c]	<20	<60	<60	1600	920	660	1300
Pentachlorobiphenyl[a]	<60	<60	<60	<60	<60	<60	2900 [c]	<20	<60	<60	1800	1200	700	1300
Hexachlorobiphenyl[a]	<60	<60	<60	<60	<60	<60	2900 [c]	2200	<60	<60	1500	1100	620	1200
Heptachlor	<20	<20	<20	<20	<20	<20	180	<20	<20	23	950	640	230	280
Lindane	<20	<20	<20	<20	<20	<20	220	<20	<20	<20	1000	490	340	<20
Toxaphene[a]	<60	<60	<60	<60	<60 [c]	<60 [c]	<60 [c]	<20	<20	<60	1300	920	750	1100
Phenols														
2,4-Dimethylphenol	<20	<20	<20	<20	2600	2700	800	<20	<20	<20	800	500	270	<20
Phenol	<20	<20	3900[b]	1600	4500 [b]	4600 [b]	1400	450	28	88	930	490	350	180
Phthalates														
Bis(2-ethylhexyl)phthalate	<20	<20	1100	350	500	620	310	<20	51	41	59	41	34	130
Butylbenzylphthalate	57	23	7600	3500	6800 [c]	6600 [c]	2700 [c]	<20	<20	<20	590	440	120	<20
Diethylphthalate	<20	<20	<20	<20	570	740	670	<20	<20	<20	830	570	130	<20
Dimethylphthalate	<20	<20	<20	<20	1700 [c]	2100 [c]	410 [c]	<20	<20	<20	900	640	170	<20
Di-n-butylphthalate	51	49	320	110	340	230	750	1100	<20	24	550	410	120	21
Di-n-octylphthalate	<20	<20	450	<20	140	170	<20	<20	<20	<20	41	28	36	46
PAHs														
Acenaphthene	<20	<20	<20	<20	2700 [c]	2900 [c]	900 [c]	2000	<20	<20	840	600	320	<20
Anthracene	<20	<20	1700	630	1500 [c]	1700 [c]	530 [c]	1200	<20	<20	570	280	220	<20
Benzo(a)anthracene	<20	<20	690	140	2700 [c]	3100 [c]	300 [c]	<20	<20	<20	580	380	280	150
Chrysene	<20	<20	330	160	1900 [c]	2200 [c]	790 [c]	<20	<20	<20	540	460	250	130
Decafluorobiphenyl	<20	<20	1300	990	1300 [c]	1400 [c]	440 [c]	380	170	110	400	280	110	180
Fluoranthene	<20	<20	<20	<20	140 [c]	280 [c]	870 [c]	240	<20	<20	540	350	270	<20
Fluorene	<20	<20	<20	<20	2800 [c]	2800 [c]	910 [c]	2200	<20	<20	880	620	340	<20
Naphthalene	<20	<20	420	2000	4300 [b,c]	4800 [b,c]	1600 [c]	1300	<20	<20	690	440	210	<20
Phenanthrene	<20	<20	<20	200	940 [c]	1200 [c]	1400 [c]	930	<20	<20	1000	490	340	<20
Pyrene	<20	<20	<20	<20	110 [c]	270 [c]	810 [c]	220	<20	<20	370	290	220	37

(a) The single most intense isomer was used to calculate the total amount of Arochlor 1254 and toxaphene.
(b) Data system saturated. Actual value is higher than this.
(c) Apparent outlier or incongrous data.

TABLE B-3. QUANTIFICATION OF PRIORITY POLLUTANTS IN WASTEWATER SAMPLED 5/15/80 (ppb)

	W Blank SSV	INF-A SSV	INF-A SSPPVI	INF-A SSPPVII	PE-A SSV	ASE-A SSV	ASE-A SSPPVI	ASE-A SSPPVII	INF-B SSV	INF-B SSVI	PE-B SSV	PE-B SSV	ASE-B SSV	ASE-B SSV
Pesticides														
Arochlor 1254														
Tetrachlorobiphenyl[a]	<3	<3	260	310	<3	<3	230	270	36	39	50[c]	6[c]	<3	<3
Pentachlorobiphenyl[a]	<3	<3	360	330	<3	<3	270	340	43	37	44[c]	<3[c]	<3	<3
Hexachlorobiphenyl[a]	<3	<3	290	340	<3	<3	290	350	42	31	32[c]	<3[c]	<3	<3
Heptachlor	<1	<1	65	70	<1	<1	130	140	11	9	<1	<1	<1	<1
Lindane	<1	3.3	60	85	<1	<1	85	85	20	26	<1	<1	3.7	3.7
Toxaphene[a]	<3	<3	240	210	<3	<3	170	230	<3	<3	<3	<3	<3	<3
Phenols														
2,4-Dimethylphenol	<1	<1	150	140	4.8	<1	46	13	40	45	29	25	<1	<1
Phenol	<1	245	350[b]	320[b]	65	6	110	130	190	210	100	87	4.4	6
Phthalates														
Bis(2-ethylhexyl)phthalate	<1	55	170	170	37	2.3	100	120	41	32	35	24	2.7	2.2
Butylbenzylphthalate	<1	<1	110	140	13	<1	90	110	26	23	14	10	<1	<1
Diethylphthalate	<1	7	80	80	6	<1	65	47	27	39	13	11	<1	<1
Dimethylphthalate	<1	3.1	20	26	1.7	<1	13	8	8	16	2.9	<1	<1	<1
Di-n-butylphthalate	<1	26	130	130	<1	1	75	90	33	37	27	21	<1	1.4
Di-n-octylphthalate	<1	<1	120	110	1.4	<1	90	100	14	8	<1	<1	<1	<1
PAHs														
Acenaphthene	<1	<1	120	110	2.4	<1	120	120	22	25	22	19	<1	<1
Anthracene	<1	<1	80	75	<1	<1	95	95	14	17	18	16	<1	<1
Benzo(a)anthracene	<1	<1	120	110	<1	<1	95	110	12	10	13	10	<1	<1
Chrysene	<1	<1	140	80	<1	<1	80	120	12	10	12	9	<1	<1
Decafluorobiphenyl	45	32	34	36	49	110	50	65	21	30	49	39	27	33
Fluoranthene	<1	<1	100	90	<1	<1	80	100	14	15	18	16	<1	<1
Fluorene	<1	3.1	90	110	3.2	<1	120	110	21	25	13	20	<1	<1
Naphthalene	<1	65	150	150	45	<1	90	100	75	90	56	42	<1	<1
Phenanthrene	<1	4.0	110	110	2.5	<1	110	120	19	23	22	21	<1	<1
Pyrene	<1	<1	85	85	<1	<1	80	100	14	14	17	15	<1	<1

(a) The single most intense isomer was used to calculate the total amount of Arochlor 1254 and toxaphene.
(b) Data system saturated. Actual value is higher than this.
(c) Apparent outlier or incongrous data.

TABLE B-4. QUANTIFICATION OF PRIORITY POLLUTANTS IN SLUDGE SAMPLED 5/15/80 (ppb)

	Blank SSV	Blank SSV	RAS-A SSV	RAS-A SSPPVI	RAS-A SSPPVII	PS-A SSV	PS-A SSPPVI	PS-A SSPPVII	RAS-B SSV	PS-B SSV	PS-B SSV	PS-B SSVI
Pesticides												
Arochlor 1254												
Tetrachlorobiphenyl[a]	90	89	200	1300	1400	800	1700	1600	340	5800	<20[c]	3900
Pentachlorobiphenyl[a]	92	71	170	1000	980	590	2400	1400	220	7200	6600	4200
Hexachlorobiphenyl[a]	<60	<60	200	850	790	550	1900	1500	190	7400	6900	4600
Heptachlor	<20	<20	64	310	530	78	580	450	27	1400	1100	960
Lindane	<20	<20	49	470	710	<20	790	530	41	<20	<20	<20
Toxaphene[a]	<60	<60	<60	1200	1300	590	1800	1700	410	6800	5700	4200
Phenols												
2,4-Dimethylphenol	<20	<20	120	210	360	<20	450	200	<20	<20	<20	<20
Phenol	<20	<20	<20	160	290	<20	400	150	<20	190	210	140
Phthalates												
Bis(2-ethylhexyl)phthalate	39	<20	120[c]	110[c]	140[c]	74	360	190	48	1200	1100	600
Butylbenzylphthalate	28	<20	100	150	190	330	1800[c]	150[c]	27	4400	3600	2100
Diethylphthalate	120[c]	160[c]	320[c]	270[c]	270[c]	460	960	1400	210	2300	2200	2200
Dimethylphthalate	<20	<20	<20	49	95	<20	790	410	<20	<20	<20	<20
Di-n-butylphthalate	67	110	90	150	200	330	1000	150	29	2500	2100	1300
Di-n-octylphthalate	<20	<20	<20	44	61	<20	75	<20	<20	450	410	<20[c]
PAHs												
Acenaphthene	<20	<20	44	270	530	120	750	320	<20	2600	2500	1300
Anthracene	<20	<20	65	320	560	200	570	290	<20	2300	2100	1500
Benzo(a)anthracene	<20	26	61	240	400	190	930	310	25	4100	3900	1800
Chrysene	<20	33	64	210	370	220	880	250	23	3600	3400	1700
Decafluorobiphenyl	<20	<20	89	100	96	66	180	110	20	190	200	130
Fluoranthene	21	26	62	320	460	220	570	280	<20	2200	1900	1100
Fluorene	<20	<20	50	380	680	140	970	580	<20	3400	3400	1700
Naphthalene	<20	<20	20	180	290	80	620	260	<20	1100	1100	610
Phenenathrene	21	25	80	380	660	260	1100	450	<20	4700	5000	2400
Pyrene	20	20	33	100	200	120	250[c]	56[c]	<20	850	730	430

(a) The single most intense isomer was used to calculate the total amount of Arochlor 1254 and toxaphene.
(c) Apparent outlier or incongrous data.

TABLE B-5. QUANTIFICATION OF PRIORITY POLLUTANTS IN WASTEWATER SAMPLED 5/28/80 (ppb)

	INF-A SSX	INF-A SSPPXI	INF-A SSPPXII	PE-A SSX	ASE-A SSX	ASE-A SSPPXI	ASE-A SSPPXI	ASE-A SSPPXII	INF-B SSX	INF-B SSXI	PE-B SSX	ASE-B SSX	ASE-B SSX	Water Blank W/SS
Pesticides														
Arochlor 1254														
Tetrachlorobiphenyl(a)	<3	230	320	<3	<3	260	280	320	<3	9.5	65(c)	<3	<3	<3
Pentachlorobiphenyl(a)	<3	290	280	<3	<3	250	280	360	10	7	65(c)	<3	<3	<3
Hexachlorobiphenyl(a)	<3	190	190	<3	<3	270	300	250	<3(c)	19(c)	75(c)	<3	<3	<3
Heptachlor	<1	100	200	<1	<1	150	150	180	14	5.5	15	2.4	8	<1
Lindane	<1	80(c)	22(c)	<1	<1	85	90	85	<1	<1	7	2.2	5.5	<1
Toxaphene(a)	<3	180	250	<3	<3	180	200	230	<3	<3	<3	<3	<3	<3
Phenols														
2,4-Dimethylphenol	21	180(c)	1.7(c)	22	<1	1.8	1.7	3.1	40	38	90(c)	<1	<1	<1
Phenol	210	480(b,c)	70(c)	230	1.6	23	20	40	300(b)	320(b)	360(b,c)	7	27	<1
Phthalates														
Bis(2-ethylhexyl)phthalate	195(c)	120(c)	85(c)	14	1.6	90	100	120	21	16	43	3.1	7	<1
Butylbenzylphthalate	4.7	110	70	4.5	<1	75	80	100	10	8	15	<1	<1	<1
Diethylphthalate	3.1	110	120	6.5	<1	49	55	55	13	8.5	15	<1	<1	<1
Dimethylphthalate	<1	39	55	<1	<1	6.5	6.5	9	<1	2.1	<1	<1	<1	<1
Di-n-butylphthalate	6	100	110	3.9	<1	80	85	100	8.5	8.5	17	<1	1.3	<1
Di-n-octylphthalate	17	110	70	1.0	<1	75	90	110	10	8.5	23	1.2	<1	<1
PAHs														
Acenaphthene	<1	140	130	<1	<1	105	100	150	20	19	65	<1	<1	<1
Anthracene	<1	130	110	<1	<1	85	130	110	12	5.5	21	<1	<1	<1
Benzo(a)anthracene	<1	110	70	<1	<1	80	90	110	11	6.5	21	<1	<1	<1
Chrysene	<1	90	65	<1	<1	70	80	100	9	5.5	11	<1	<1	<1
Decafluorobiphenyl	20	46	65	55	40	47	50	65	30	27	80	31	100	38
Fluoranthene	<1	85	90	<1	<1	75	95	95	9.5	6.5	22	<1	<1	<1
Fluorene	2.1	140	140	1.3	<1	110	110	160	20	9	65	<1	<1	<1
Naphthalene	13	150	100	17	<1	75	80	90	35	35	85	<1	<1	<1
Phenanthrene	2.0	130	120	2.2	<1	110	100	190	22	11	33	<1	<1	<1
Pyrene	<1	90	80	<1	<1	75	90	95	9.5	6.5	22	<1	1.3	<1

(a) The single most intense isomer was used to calculate the total amount of Arochlor 1254 and toxaphene.
(b) Data system saturated. Actual value is higher than this.
(c) Apparent outlier or incongrous data.

TABLE B-6. QUANTIFICATION OF PRIORITY POLLUTANTS IN SLUDGE SAMPLED 5/28/80 (ppb)

	Blank SSX	RAS-A SSX	RAS-A SSPPXI	RAS-A SSPPXI	RAS-A SSPPXII	PS-A SSX	PS-A SSPPXI	PS-A SSPPXII	RAS-B SSX	PS-B SSX	PS-B SSXI
Pesticides											
Arochlor 1254											
Tetrachlorobiphenyl[a]	<60	330	2500	3000	2200	<60	970	1300	1600	6200	<60[c]
Pentachlorobiphenyl[a]	<60	250	1900	2800	2100	<60	1000	1500	1300	5800	11000
Hexachlorobiphenyl[a]	<60	210	1700	2600	2000	<60	850	1200	1400	6000	9600
Heptachlor	<20	64	1000	1100	1000	<20	240	340	390	1500	<20[c]
Lindane	<20	87	1100	1100	1100	800[c]	300[c]	770[c]	130	820	1106
Toxaphene[a]	<60	200	2600	2900	2400	<60	1200	1300	1300	5200	<60[c]
Phenols											
2,4-Dimethylphenol	<20	<20	510	450	620	<20	220	590	<20	<20	<20
Phenol	<20	<20	350	290	370	190	20[c]	570[c]	<20	<20[c]	180[c]
Phthalates											
Bis(2-ethylhexyl)phthalate	27	<20	110	220	210	8600[b]	6700[b]	12000[b]	140	1000[c]	10000[b,c]
Butylbenzylphthalate	45	76	460	900	610	1400[c]	1000[c]	1500[c]	42	1500	3400
Diethylphthalate	<20	200	860	710	760	<20	1100	2000	220	1400	260
Dimethylphthalate	<20	25	600	510	560	<20	380	690	<20	79	<20
Di-n-butylphthalate	53	110	710	790	590	310	390	630	49	1000[c]	<20[c]
Di-n-octylphthalate	<20	<20	62	130	110	180	380	250	53	38[c]	1100[c]
PAHs											
Acenaphthene	<20	73	790	630	690	<20	450	780	30	1000	2900
Anthracene	<20	93	980	990	840	<20	150	540	50	1500	2300
Benzo(a)anthracene	<20	51	500	950	640	76	500	700	140	1800	6600
Chrysene	<20	58	520	960	540	57	340	620	130	1800	4200
Decafluorobiphenyl	<20	130	320	250	290	310	140	250	220	79	340
Fluoranthene	23	81	760	1100	650	110	290	460	71	1700	2800
Fluorene	<20	84	930	780	920	140	530	890	<20	1300	4700
Naphthalene	<20	39	430	380	450	390	390	790	<20	210	880
Phenanthrene	27	110	1100	1000	900	<20	280	1100	50	1700	2400
Pyrene	<20	79	380	560	280	35[c]	110[c]	110[c]	60	560	1100

(a) The single most intense isomer was used to calculate the total amount of Arochlor 1254 and toxaphene.
(b) Data system saturated. Actual value is higher than this.
(c) Apparent outlier or incongrous data.

TABLE B-7. SUMMARY OF CONTROL SEQUENCE A SAMPLES OF MAY 4, 15, 28, 1980 (ppb)

	Influent	Primary Effluent	Activated Sludge Effluent	Primary Sludge	Return Activated Sludge
PESTICIDES/PCBs					
Arochlor 1254	<2.8	<2.8	<2.2	<260	<170
Heptachlor	<1.0	<1	<1	<39	<50
Lindane	<1.8	<1	<1	<290	<52
Toxaphene	<2.8	<2.8	<2.2	<240	<110
PHENOLS					
2,4-Dimethylphenol	<7.9	<16	<0.8	<20	<53
Phenol	220	320	5.5	<990	<33
PHTHALATES					
Bis(2-ethylhexyl)phthalate	92	27	2.0	3100	<62
Butylbenzylphthalate	<3.3	7.7	<0.8	2400	<50
Diethylphthalate	4.4	7.2	<1	<120	<130
Dimethylphthalate	<1.5	<4.5	<0.8	<20	<22
Di-n-butylphthalate	12	<3.2	<0.9	260	<33
Di-n-octylphthalate	<6.5	<1.3	<0.8	<140	<20
POLYNUCLEAR AROMATIC HYDROCARBONS					
Acenaphthene	<0.8	<2.1	<0.8	<53	<46
Anthracene	<1.0	<1	<1	<460	<59
Benzo(a)anthracene	<1.0	<1	<1	230	<44
Chrysene	<0.8	<0.8	<0.8	170	<47
Decafluorobiphenyl	-	-	-	-	-
Fluoranthene	<0.8	<0.8	<0.8	<109	<39
Fluorene	<1.9	<0.9	<0.8	<100	<51
Naphthalene	44	39	<1.4	1200	<26
Phenanthrene	5.3	<2.1	<0.8	<130	<53
Pyrene	<0.8	<0.8	<0.8	<52	<37

TABLE B-8. SUMMARY OF SPIKED SEQUENCE B SAMPLES OF MAY 4, 15, 28, 1980 (ppb)

	Influent	Primary Effluent	Activated Sludge Effluent	Primary Sludge	Return Activated Sludge
PESTICIDES/PCBs					
Arochlor 1254	27	55	<2.8	15,000	3,200
Heptachlor	16	<9.4	<2.6	<1,300	480
Lindane	<20	<7.9	6.2	<330	<100
Toxaphene	<8.1	<2.8	<2.8	<9,300	2,600
PHENOLS					
2,4-Dimethylphenol	64	89	<0.8	<20	<20
Phenol	<390	<360	28	1,700	<190
PHTHALATES					
Bis(2-ethylhexyl)phthalate	40	43	4.8	<5,500	4,800
Butylbenzylphthalate	16	14	<0.8	<2,500	<220
Diethylphthalate	31	19	<0.8	<1,300	<260
Dimethylphthalate	30	<6.2	<0.8	<49	<20
Di-n-butylphthalate	24	23	<1.2	4,100	230
Di-n-octylphthalate	19	<24	<1.1	<4,200	<860
POLYNUCLEAR AROMATIC HYDROCARBONS					
Acenaphthene	34	45	<0.8	4,200	<38
Anthracene	26	27	<0.8	6,700	<42
Benzo(a)anthracene	17	23	<0.8	<8,400	330
Chyrsene	16	17	<0.8	<6,800	310
Decafluorobiphenyl	-	-	-	-	-
Fluoranthene	18	28	<0.8	7,400	<59
Fluorene	32	47	<0.8	4,900	<20
Naphthalene	108	109	<0.8	2,800	<20
Phenanthrene	31	35	<0.8	5,200	<40
Pyrene	20	28	<1.2	14,000	<160

TABLE B-9. TOTAL RESIDUE FROM WASTEWATER EXTRACTS

Sample	Total Residue (mg)	Sample	Total Residue (mg)	Sample	Total Residue (mg)
INF-A SSI	40	INF-A SSV	150	INF-A SSX	58
INF-A SSII	43				
INF-A SSPPIII	54	INF-A SSPPVI	190	INF-A SSPPXI	73
INF-A SSPPIV	52	INF-A SSPPVII	130	INF-A SSPPXII	11
SSE-A SSI	58				
GCE-A SSI	55				
PE-A SSI	42	PE-A SSV	100	PE-A SSX	73
PE-A SSII	44				
PE-A SSPPIII	49				
PE-A SSPPIV	46				
ASE-A SSI	11	ASE-A SSV	10	ASE-A SSX	8
ASE-A SSII	11				
ASE-A SSPPIII	14	ASE-A SSPPVI	17	ASE-A SSPPXI	12
ASE-A SSPPIV	15	ASE-A SSPPVII	16	ASE-A SSPPXII	12
INF-B SSI	43	INF-B SSV	160	INF-B SSX	88
INF-A SSII	41	INF-B SSVI	180	INF-B SSXI	87
SSE-B SSI	49				
GCE-B SSI	75				
PE-B SSI	58	PE-B SSV	110	PE-B SSX	62
ASE-B SSI	10	ASE-B SSV	6	ASE-B SSX	7
Blank	0	Blank	0	Blank	0

TABLE B-10. TOTAL RESIDUE FROM SLUDGE EXTRACTS

Sample	Total Residue (mg) Before Clean-up	Total Residue (mg) After Clean-up	Sample	Total Residue (mg) Before Clean-up	Total Residue (mg) After Clean-up	Sample	Total Residue (mg) Before Clean-up	Total Residue (mg) After Clean-up
PS-A SSI	850	240	PS-A SSV	350	13	PS-A SSX	860	190
PS-A SSII	860	230	PS-A SSPPVI	230	83			
PS-A SSPPIII	830	290	PS-A SSPPVII	330	24	PS-A SSPPXI	840	70
PS-A SSPPIV	770	210				PS-A SSPPXII	850	72
			RAS-A SSV	79	19			
RAS-A SSI	96	20				RAS-A SSX	68	18
RAS-A SSII	94	20	RAS-A SSPPVI	72	10			
RAS-A SSPPIII	97	32	RAS-A SSPPVII	69	16	RAS-A SSPPXI	62	9
RAS-A SSPPIV	110	16				RAS-A SSPPXII	61	11
			PS-B SSV	830	78			
PS-B SSI	770	320	PS-B SSVI	900	47	PS-B SSX	900	18
						PS-B SSXI	860	160
RAS-B SSI	73	19	RAS-B SSV	33	6			
						RAS-B SSX	34	7
Blank	.6	---	Blank	3	--			
						Blank	3	3

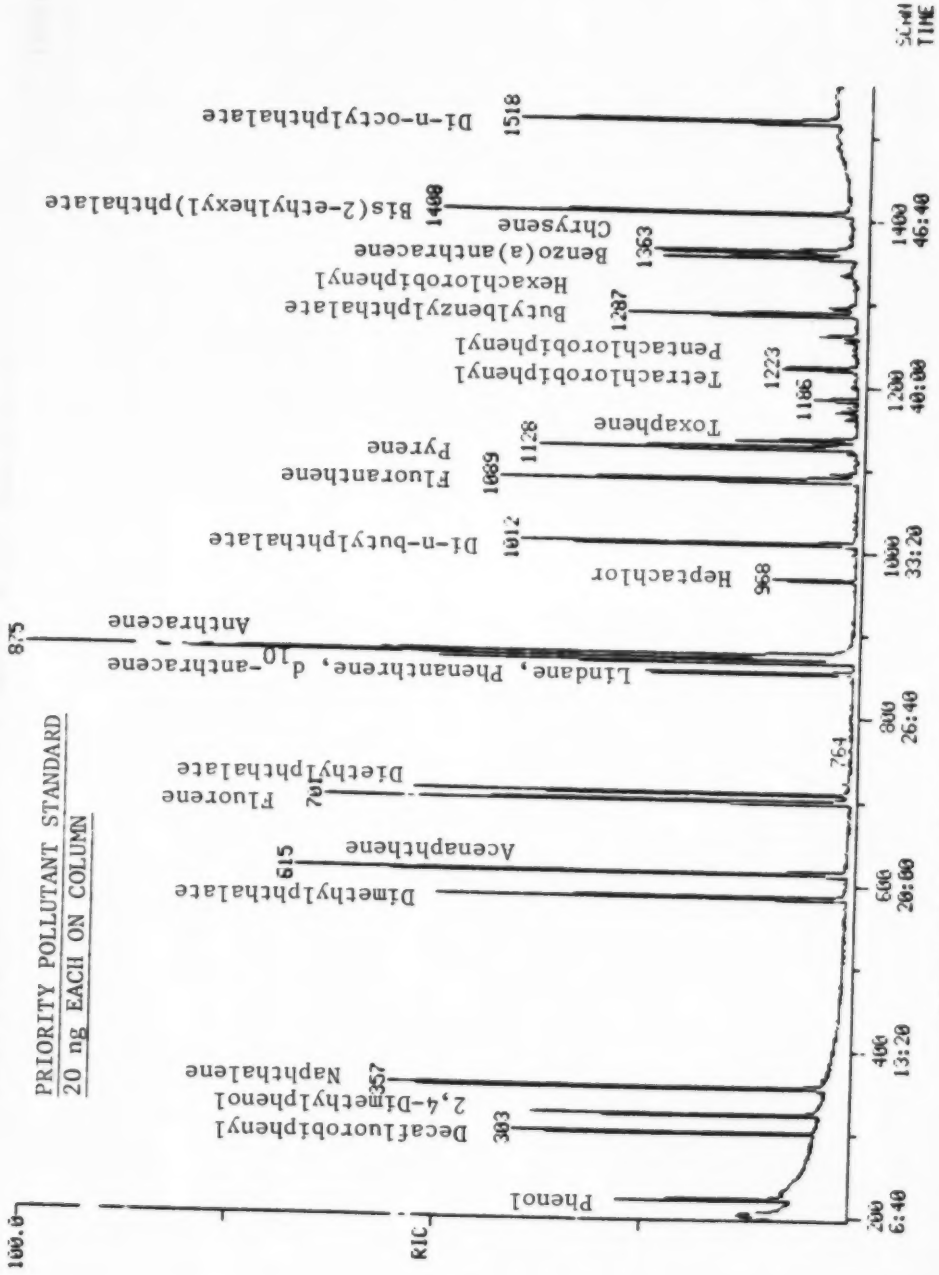


Figure B-1. Reconstructed ion chromatogram of priority pollutant standard (20 ng on column.

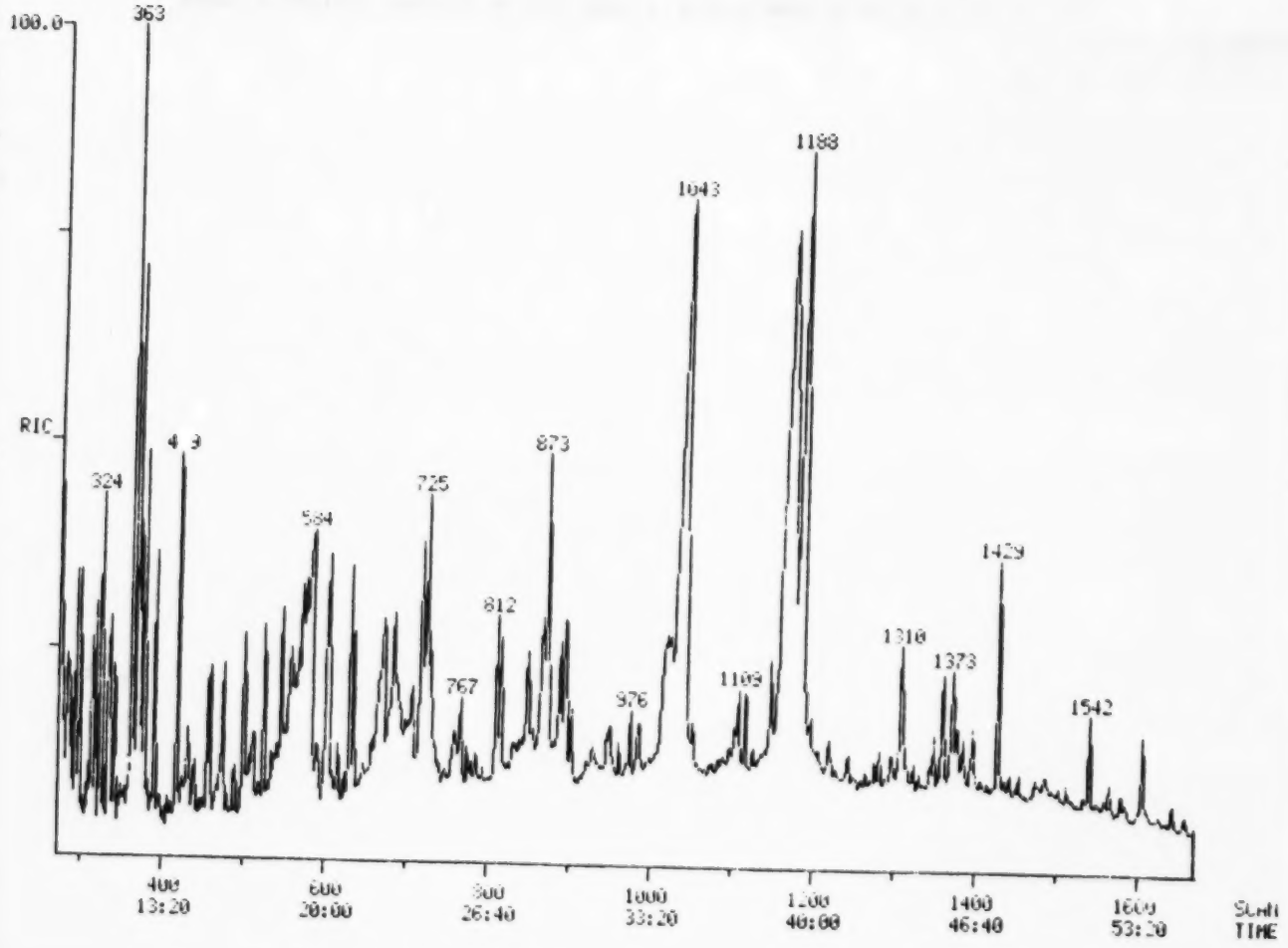


Figure B-2. Reconstructed ion chromatogram of INF-B SSI.

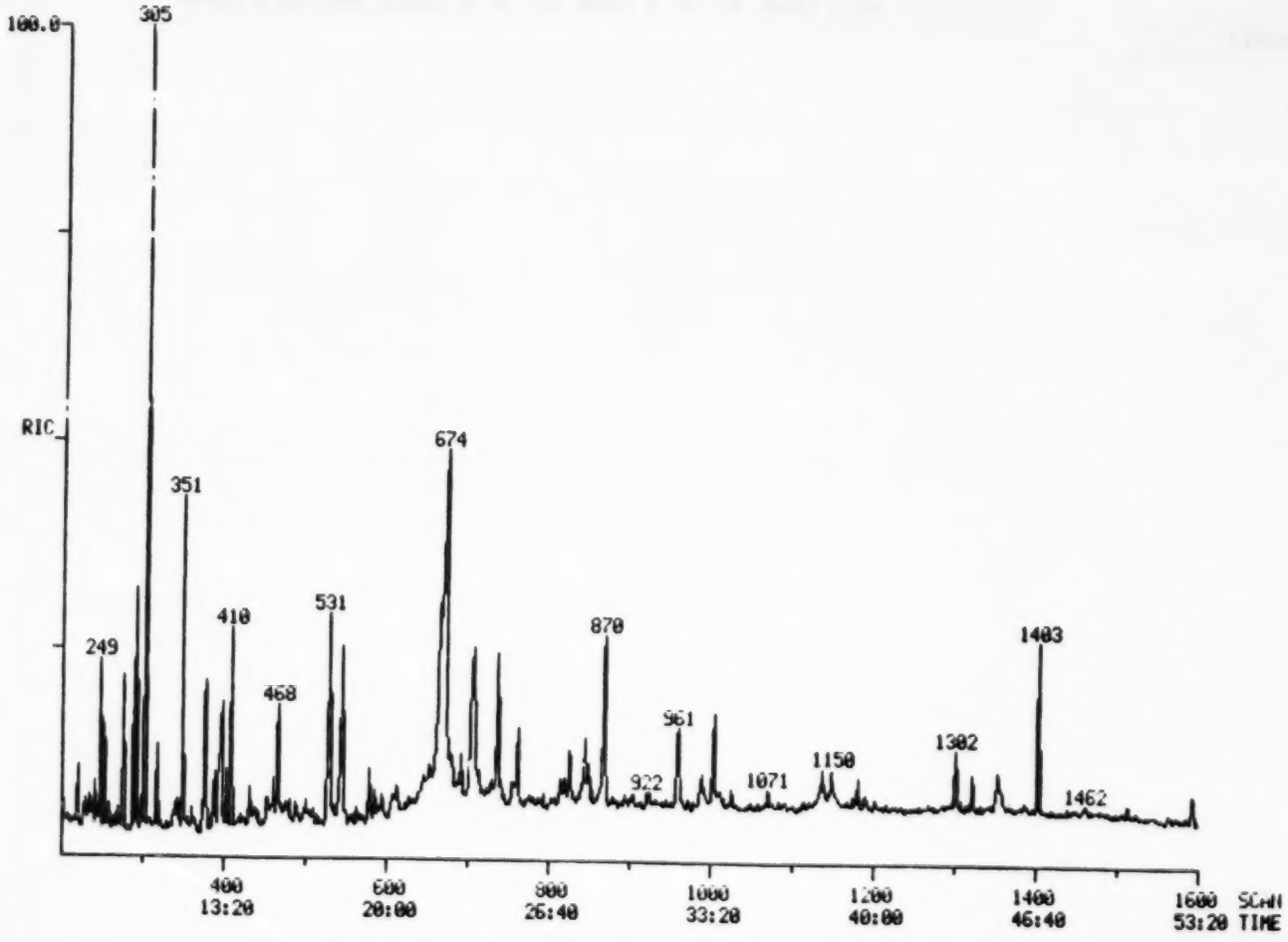


Figure B-3. Reconstructed ion chromatogram of ASE-B SSI.

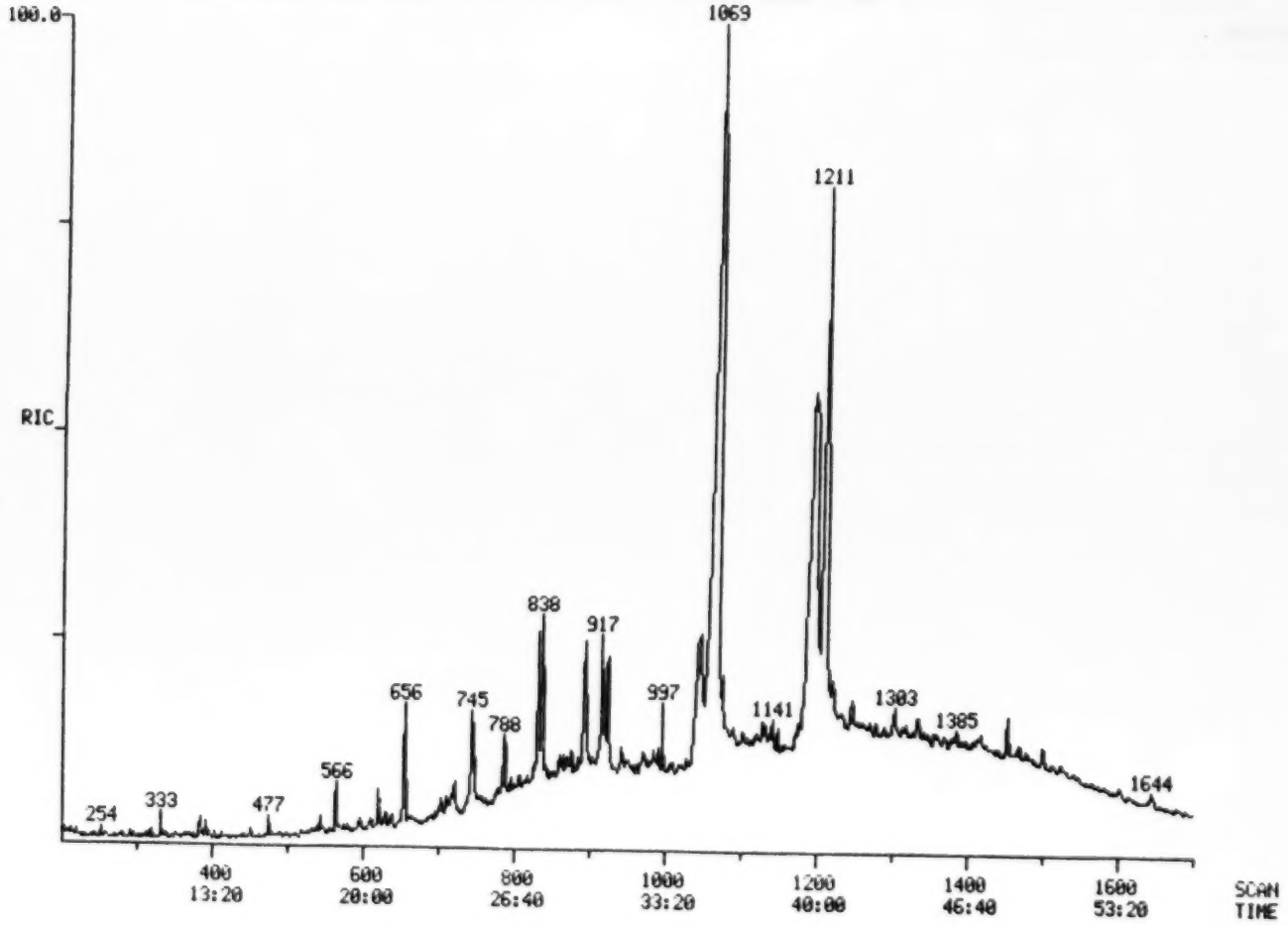


Figure B-4. Reconstructed ion chromatogram of RAS-A SSV.

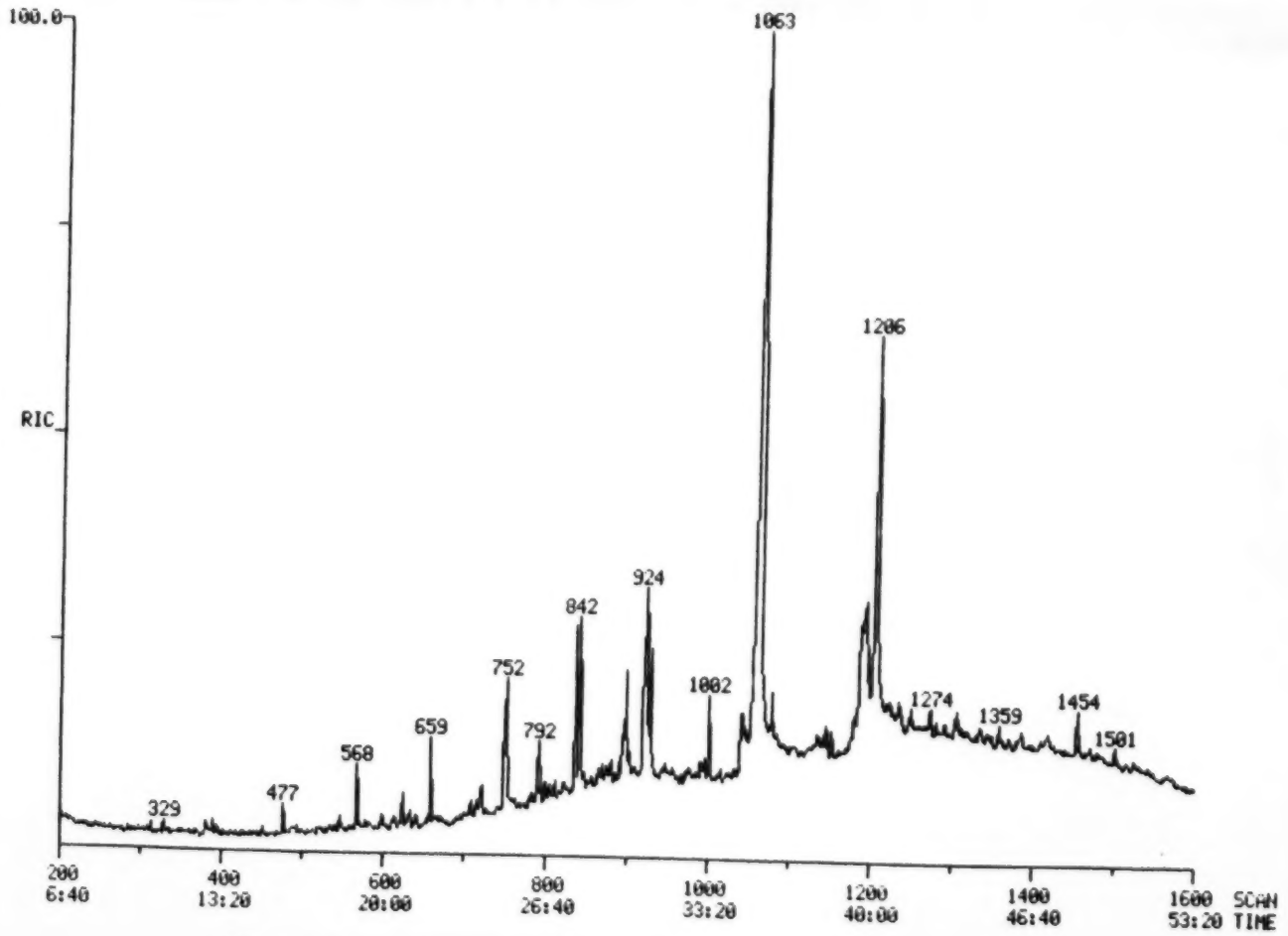


Figure B-5. Reconstructed ion chromatogram of RAS-B SSV.

APPENDIX C

QUALITY ASSURANCE DATA

TABLE C-1. QUANTIFICATION OF PROCESS BLANKS (ppb)

	Water Blank SSI	Water Blank SSV	Water Blank SSX	Sludge Blank SSI	Sludge Blank SSI	Sludge Blank SSV	Sludge Blank SSV	Sludge Blank SSX
PESTICIDES/PCBs								
Arochlor 1254								
Tetrachlorobiphenyl[a]	<3	<3	<3	<60	<60	90	89	<60
Pentachlorobiphenyl[a]	<3	<3	<3	<60	<60	92	71	<60
Hexachlorobiphenyl[a]	<3	<3	<3	<60	<60	<60	<60	<60
Heptachlor	<1	<1	<1	<20	<20	<20	<20	<20
Lindane	<1	<1	<1	<20	<20	<20	<20	<20
Toxaphene[a,b]	<3	<3	<3	<60	<60	<60	<60	<60
PHENOLS								
2,4-Dimethylphenol	<1	<1	<1	<20	<20	<20	<20	<20
Phenol	<1	<1	<1	<20	<20	<20	<20	<20
PHTHALATES								
Bis(2-ethylhexyl)phthalate	<1	<1	<1	<20	<20	39	<20	27
Butylbenzylphthalate	<1	<1	<1	57	23	28	<20	45
Diethylphthalate	<1	<1	<1	<20	<20	120[b]	160[b]	<20
Dimethylphthalate	<1	<1	<1	<20	<20	<20	<20	<20
Di-n-butylphthalate	<1	<1	<1	51	49	67	110	53
Di-n-octylphthalate	<1	<1	<1	<20	<20	<20	<20	<20
PAHs								
Acenaphthene	<1	<1	<1	<20	<20	<20	<20	<20
Anthracene	<1	<1	<1	<20	<20	<20	<20	<20
Benzo(a)anthracene	<1	<1	<1	<20	<20	<20	26	<20
Chrysene	<1	<1	<1	<20	<20	<20	33	<20
Decafluorobiphenyl	13	45	38	<20	<20	<20	<20	<20
Fluoranthene	<1	<1	<1	<20	<20	21	26	23
Fluorene	<1	<1	<1	<20	<20	<20	<20	<20
Naphthalene	<1	<1	<1	<20	<20	<20	<20	<20
Phenanthrene	<1	<1	<1	<20	<20	21	25	27
Pyrene	<1	<1	<1	<20	<20	20	20	<20

(a) The single most intense isomer was used to calculate the total amount of Arochlor 1254 and toxaphene.

(b) Apparent outliers.

TABLE C-2. AVERAGE RESPONSE FACTORS (R_f) OBTAINED FROM CALIBRATION CURVE

	Std A, 2.5 µg/ml	Std B, 10 µg/ml	Std C, 50 µg/ml	Std D, 100 µg/ml	Std E, 250 µg/ml	Mean
PESTICIDES/PCBs						
Arochlor 1254						
Tetrachlorobiphenyl	0.010	0.016	0.016	0.013	0.015	0.014
Pentachlorobiphenyl	0.017	0.023	0.021	0.020	0.025	0.021
Hexachlorobiphenyl	0.0062	0.0092	0.0078	0.0074	0.0086	0.0081
Heptachlor	0.065	0.083	0.091	0.092	0.10	0.087
Lindane	0.085	0.10	0.10	0.088	0.10	0.097
Toxaphene	0.0032	0.0043	0.0036	0.0032	0.0036	0.0036
PHENOLS						
2,4-Dimethylphenol	0.27	0.37	0.37	0.30	0.31	0.32
Phenol	0.55	0.64	0.63	0.42	0.42	0.53
PHTHALATES						
Bis(2-ethylhexyl)phthalate	0.34	0.52	0.60	0.57	0.62	0.53
Butylbenzylphthalate	0.23	0.33	0.40	0.38	0.45	0.36
Diethylphthalate	0.62	0.91	0.97	0.68	0.69	0.78
Dimethylphthalate	0.66	0.92	0.92	0.66	0.66	0.76
Di-n-butylphthalate	0.64	1.2	1.4	0.91	0.85	0.99
Di-n-octylphthalate	0.48	0.74	0.59	0.77	0.94	0.71
PAHs						
Acenaphthene	0.52	0.68	0.69	0.53	0.53	0.59
Anthracene	0.87	1.1	1.1	0.80	0.75	0.92
Benzo(a)anthracene	0.23	0.33	0.41	0.45	0.55	0.39
Chrysene	0.24	0.45	0.53	0.50	0.56	0.46
Decafluorobiphenyl	0.34	0.48	0.45	0.30	0.33	0.38
Fluoranthene	0.56	0.85	0.88	0.75	0.75	0.76
Fluorene	0.52	0.76	0.78	0.60	0.61	0.65
Naphthalene	1.3	1.6	1.4	0.75	0.68	1.15
Phenanthrene	0.75	1.0	1.1	0.79	0.75	0.88
Pyrene	0.56	0.85	0.90	0.76	0.76	0.77

TABLE C-3. RECOVERY OF PRIORITY POLLUTANTS FROM DISTILLED WATER (%)

	Replicates									
	1	2	3	4	5	6	7	8	Mean	rsd
PESTICIDES/PCBs										
Arochlor 1254										
Tetrachlorobiphenyl	104				108	113	121	107	111	6.0
Pentachlorobiphenyl	66				76	72	77	73	73	5.9
Hexachlorobiphenyl	62	128	105		49	67	66	63	77	37
Heptachlor	59	58	70	70	58	59	66	70	64	9.1
Lindane	72	68	77	71	61	66	71	71	70	6.8
Toxaphene	157				60	75	101	88	96	39
PHENOLS										
2,4-Dimethylphenol	55	57	63	62	54	46	59	62	57	9.9
Phenol	66	61	72	68	65	57	67	77	66	9.3
PHTHALATES										
Bis(2-ethylhexyl)phthalate	45	66	72	88	49	57	57	55	61	23
Butylbenzylphthalate	65				59	70	76	73	69	9.8
Diethylphthalate	61	65	72	71	66	67	71	68	68	5.5
Dimethylphthalate	59	70	78	75	68	67	73	70	70	8.2
Di-n-butylphthalate	74	94	114	106	85	107	114	107	100	14
Di-n-octylphthalate	56	93	102	113	58	88		71	83	26
PAHs										
Acenaphthene	56	64	70	68	58	54	60	63	62	9.2
Anthracene	68	67	78	78	71	74	75	74	73	5.7
Benzo(a)anthracene	58	78	85	54	58	63	71	73	68	16
Chrysene	66		137		113	135	162	122	122	26
Fluoranthene	68	70	80	85	71	74	77	81	76	7.9
Fluorene	73	81	99	95	77	79	87	81	84	11
Naphthalene	48	47	56	51	49	43	49	59	50	10
Phenanthrene	68	68	78	79	73	75	77	76	74	5.7
Pyrene	68	74	83	96	73	75	79	80	79	11
Mean Recovery									76	

TABLE C-4. RECOVERY OF PRIORITY POLLUTANT SPIKE FROM WASTEWATER (%)

	INF-A III	INF-A IV	PE-A III	PE-A III	PE-A IV	ASE-A III	ASE-A IV	INF-A VI	INF-A VII	ASE-A VI	ASE-A VII	INF-A XI	INF-A XII	ASE-A XI	ASE-A XI	ASE-A XII	Mean	rsd
Pesticides																		
Arochlor 1254																		
Tetrachlorobiphenyl	52	76	72	66	72	66	97	61	71	53	63	53	74	61	64	75	67	11
Pentachlorobiphenyl	59	79	69	69	69	66	69	84	77	62	78	67	66	57	66	84	70	8.2
Hexachlorobiphenyl	55	72	62	57	59	52	57	67	78	67	80	45	45	63	69	59	62	10
Heptachlor	76	130	120	130	140	170	200	45	48	90	100	69	140	100	110	130	110	41
Lindane	55	97	110	200	130	83	90	39	56	59	59	55	15	59	62	59	77	43
Toxaphene	62	110	97	69	72	76	83	56	48	39	53	43	57	41	47	53	63	20
Phenols																		
2,4-Dimethylphenol	52	93	90	93	97	27	29	100	97	31	8.6	100	0	1.2	1.1	2.1	52	43
Phenol						87	100	76	55	75	89		0	14	13	26	54	37
Phthalates																		
Bis(2-ethylhexyl)phthalate	58	99	110	110	110	140	160	83	83	67	81	0	0	61	68	82	82	42
Butylbenzylphthalate	76	120	120	110	120	140	160	79	100	62	76	73	45	52	55	72	91	34
Diethylphthalate	38	72	64	61	78	55	69	50	50	45	32	74	84	33	38	38	55	17
Dimethylphthalate	22	48	34	33	55	3.2	4.5	12	15	9.0	5.2	27	38	4.5	4.5	6.2	20	17
Di-n-butylphthalate	46	81	110	84	110	140	140	72	75	52	62	65	75	55	59	69	81	29
Di-n-octylphthalate	76	120	130	120	130	160	180	83	76	62	69	68	37	52	62	76	94	41
PAHs																		
Acenaphthene	45	48	82	79	85	97	100	83	79	83	86	100	90	72	69	110	82	17
Anthracene	45	72	79	72	83	83	93	55	52	66	66	59	79	59	90	76	71	14
Benzo(a)anthracene	69	97	97	93	90	100	110	86	76	66	79	76	48	55	62	76	80	17
Chrysene	59	48	79	170	76	79	93	66	55	55	86	62	45	48	55	69	72	30
Decafluorobiphenyl	29	45	50	45	49	50	55	34	36	50	65	46	65	47	50	65	49	10
Fluoranthene	48	48	83	76	79	93	100	69	62	55	72	59	62	52	66	66	68	15
Fluorene	52	48	85	81	88	100	110	60	77	83	76	95	95	76	76	110	82	18
Naphthalene	14	56	76	34	69	99	110	62	59	62	69	94	63	52	55	62	65	23
Phenanthrene	46	78	85	74	85	97	110	73	73	76	83	88	85	79	79	130	84	18
Pyrene	48	79	83	79	79	90	100	66	59	55	69	62	55	52	62	66	69	14

[% recovery = $\frac{M - P}{S} \times 100$] M = value measured in spiked sample extract, P = amount originally present as measured in unspiked sample extract, S = amount spiked into the sample.

TABLE C-5. RECOVERY OF PRIORITY POLLUTANT SPIKE FROM SLUDGE (%)

	PS-A III	PS-A III	PS-A IV	RAS-A III	RAS-A III	RAS-A IV	RAS-A VI	RAS-A VII	PS-A VI	PS-A VII	RAS-A XI	RAS-A XI	RAS-A XII	PS-A XI	PS-A XII	Mean	rsd
Pesticides																	
Arochlor 1254																	
Tetrachlorobiphenyl	0	0	52	38	22	16	26	29	21	19	52	64	45	23	31	29	18
Pentachlorobiphenyl	0	0	69	43	29	17	20	19	43	19	39	73	44	24	36	32	21
Hexachlorobiphenyl	0	0	69	36	26	15	15	14	32	23	35	57	43	20	29	28	19
Heptachlor	0	0	13	68	46	46	18	33	36	27	67	74	67	17	24	36	25
Lindane	0	0	16	71	35	24	30	47	56	38	72	72	72	0	0	36	29
Toxaphene	0	0	0	31	22	18	29	31	29	26	57	64	52	29	31	28	19
Phenols																	
2,4-Dimethylphenol	190	190	63	57	36	19	6.4	17	32	14	36	32	44	16	42	53	58
Phenol	130	130	0	62	31	21	11	21	29	11	25	21	26	0	27	36	41
Phthalates																	
Bis(2-ethylhexyl)phthalate	0	0	0	0	0	0	0	1.4	20	8.3	7.9	16	15			5.3	7.4
Butylbenzylphthalate	89	75	0	42	31	8.6	3.6	6.4	100	0	27	59	38	0	7.1	32	34
Diethylphthalate	41	53	48	59	41	9.3	0	0	36	67	47	36	40	79	140	46	34
Dimethylphthalate	120	150	29	64	46	12	3.5	6.8	56	29	41	35	38	27	49	47	40
Di-n-butylphthalate	8.9	1.1	38	34	24	3.3	4.3	7.9	48	0	43	49	34	5.7	23	22	18
Di-n-octylphthalate	0	0	0	2.9	2.0	2.6	3.1	4.4	5.4	0	4.4	9.3	7.9	14	5.0	4.1	4.0
PAHs																	
Acenaphthene	190	210	64	60	43	23	16	35	45	14	51	40	44	32	56	62	58
Anthracene	24	38	0	41	20	16	18	35	26	6.4	63	64	53	11	39	30	20
Benzo(a)anthracene	160	190	0	41	27	20	13	24	53	8.6	32	64	42	30	45	50	54
Chrysene	120	140	39	39	33	18	10	22	47	2.1	33	64	34	20	40	44	38
Decafluorobiphenyl	130	140	44	40	28	11	10	9.6	18	11	32	25	29	14	25	38	41
Fluoranthene	10	20	62	39	25	19	18	28	25	4.3	49	73	41	13	25	30	19
Fluorene	200	200	65	63	44	24	24	45	59	31	60	50	60	28	54	67	56
Naphthalene	86	120	0	49	31	15	11	19	39	13	28	24	29	0	29	33	32
Phenanthrene	53	71	86	71	35	26	21	41	60	14	71	64	56	20	79	51	24
Pyrene	7.9	19	58	26	21	16	4.8	12	9.3	0	22	34	14	5.4	5.4	17	15

$\left[\% \text{ Recovery} = \frac{M - P}{S} \times 100\right]$ M = value measured in spiked sample extract, P = amount originally present as measured in unspiked sample extract, S = amount spiked into sample

TABLE C-6. OBSERVED RECOVERY IN WASTEWATER SAMPLES (%)

	INFLUENT				ACTIVATED SLUDGE EFFLUENT			
	5/4/80	5/15/80	5/28/80	$\bar{x}$	5/4/80	5/15/80	5/28/80	$\bar{x}$
PESTICIDES/PCBs								
Arochlor 1254	66	75.6	60.1	67.2	69	69.6	67.1	68.6
Heptachlor	105	48.2	107.1	86.8	187	98.2	120.6	153.3
Lindane	77	51.8	36.3	55.0	87	60.7	61.6	69.8
Toxaphene	95	54.2	51.8	67.0	80	47.6	50.3	59.3
PHENOLS								
2,4-Dimethylphenol	72	103.6	51.7	75.8	28	20.7	1.7	16.8
Phenol	17	67.9	48.2	44.4	97	89.3	21.9	69.4
PHTHALATES								
Bis(2-ethylhexyl)phthalate	79	85.7	-64.3(a)	33.5	157	77.5	76.8	103.8
Butylbenzylphthalate	98	92.9	60.9	83.9	152	71.4	65.2	96.2
Diethylphthalate	55	52.1	81.7	62.9	63	40.0	38.1	47.0
Dimethylphthalate	36	14.1	33.6	27.9	2.6	7.3	5.6	5.2
Di-n-butylphthalate	64	76.1	72.5	70.9	145	58.9	65.2	89.7
Di-n-octylphthalate	99	82.1	53.9	78.3	175	67.9	68.8	103.9
POLYNUCLEAR AROMATIC HYDROCARBONS								
Acenaphthene	65	83.9	98.2	82.4	101	87.5	92.0	93.5
Anthracene	59	55.4	71.4	61.9	90	67.9	77.7	78.5
Benzo(a)anthracene	84	83.9	64.3	77.4	107	75.0	69.6	83.9
Chyrsene	72	62.5	55.4	63.3	87	73.2	62.5	74.2
Decafluorobiphenyl	--	--	--	--	--	--	--	--
Fluoranthene	66	67.9	62.5	65.5	100	65.2	46.4	70.5
Fluorene	69	71.0	98.5	79.5	107	82.1	98.2	95.8
Naphthalene	31	62.5	81.8	58.4	105	67.9	59.9	77.6
Phenanthrene	63	75.7	89.6	76.1	105	82.1	107.1	194.2
Pyrene	65	64.3	60.7	63.3	115	64.3	63.4	80.9

(a) Apparent negative recovery resulting from high background levels.

TABLE C-7. OBSERVED RECOVERIES IN SLUDGE SAMPLES (%)

	Primary Sludge				Return Activated Sludge			
	5/4/80	5/15/80	5/28/80	$\bar{x}$	5/4/80	5/15/80	5/28/80	$\bar{x}$
PESTICIDES/PCBs								
Arochlor 1254	27.1	26.3	27.1	26.8	22.9	27.6	47.5	32.7
Heptachlor	<7.1	31.2	20.7	<19.7	36.6	25.4	68.6	43.5
Lindane	<8.6	47.1	-18.9(a)	<24.9	38.8	38.6	72.4	49.9
Toxaphene	<4.3	27.6	29.8	<20.3	66.4	29.8	56.5	50.9
PHENOLS								
2,4-Dimethylphenol	126.1	23.2	28.9	59.4	32.9	11.8	39.3	28.0
Phenol	16.1	10.6	7.5	14.4	33.7	16.1	24.6	24.8
PHTHALATES								
Bis(2-ethylhexyl)phthalate	-207.1(a)	14.4	53.4	-46.4(a)	-0.3(a)	0.36	13.4	4.5
Butylbenzylphthalate	-60.7(a)	46.1	-10.7(a)	-8.4(a)	22.7	5.0	43.9	23.9
Diethylphthalate	47.4	74.3	110.7	77.5	29.6	-3.4(a)	40.9	22.4
Dimethylphthalate	82.5	42.9	38.2	37.9	33.6	5.1	38.0	25.6
Di-n-butylphthalate	21.6	17.6	14.3	17.8	21.4	6.2	43.8	23.8
Di-n-octylphthalate	-4.3(a)	3.4	9.6	2.9	2.5	3.8	7.4	4.7
POLYNUCLEAR AROMATIC HYDROCARBONS								
Acenaphthene	132.1	29.6	43.9	68.5	58.6	25.4	40.2	29.3
Anthracene	-7.1(a)	16.4	24.6	11.3	23.1	26.8	58.5	36.1
Benzo(a)anthracene	84.6	20.7	37.4	47.6	27.1	18.5	45.1	30.2
Chrysene	83.9	24.6	30.2	46.2	26.8	16.1	41.6	28.2
Decafluorobiphenyl	-17.8(a)	--	--	--	6.1	--	--	--
Fluoranthene	36.8	16.3	18.9	24.0	25.6	23.4	52.3	33.8
Fluorene	133.5	45.4	40.7	73.2	39.1	34.3	57.4	43.6
Naphthalene	-1.8(a)	25.7	14.3	12.7	27.7	15.4	27.8	23.6
Phenanthrene	87.5	35.1	49.3	57.3	38.8	31.4	63.7	34.2
Pyrene	35.7	2.4	5.4	14.5	19.6	8.4	21.1	16.4

(a) Apparent negative recoveries resulting from high background and interferences.

TABLE C-8. QUANTIFICATION OF STANDARD SOLUTIONS (µg/ml)

	A	A	A	A	A	B	B	B	B	B	B
PESTICIDES/PCBs											
Arochlor 1254											
Tetrachlorobiphenyl[a,b]	5.4	<5	8.4	6.7	<5	30	33	30	35	31	24
Pentachlorobiphenyl[a,b]	4.8	<5	8.4	5.8	<5	36	38	25	40	26	23
Hexachlorobiphenyl[a,b]	5.4	<5	7.0	6.6	<5	33	30	40	33	44	28
Heptachlor	2.8	3.2	1.4	1.5	<2	11	10	9.8	11	10	3.8
Lindane	1.9	2.9	1.9	1.8	<2	9.7	11	10	12	11	6.2
Toxaphene[a,b]	<5	<5	5.1	<5	<5	39	42	33	47	37	25
PHENOLS											
2,4-Dimethylphenol	2.7	1.7	1.8	<5	<2	13	11	9.9	12	11	7.6
Phenol	3.5	<2	2.2	<5	<2	14	9.4	10	9.8	11	7.1
PHTHALATES											
Bis(2-ethylhexyl)phthalate	3.1	3.2	2.1	1.1	<2	18	14	6.4	15	6.9	5.3
Butylbenzylphthalate	3.5	2.7	1.5	1.1	<2	17	13	9.2	13	9.3	5.2
Diethylphthalate	2.6	2.4	1.9	1.5	<2	12	11	9.7	12	11	7.1
Dimethylphthalate	2.6	2.2	2.1	1.8	<2	12	11	9.4	12	10	7.3
Di-n-butylphthalate	2.5	2.1	1.8	1.2	<2	14	11	9.2	13	11	5.8
Di-n-octylphthalate	3.7	2.9	2.0	1.0	<2	22	14	10	16	12	5.7
PAHs											
Acenaphthene	2.3	2.5	2.1	1.8	<2	11	10	10	11	11	7.4
Anthracene	2.8	2.3	2.1	2.4	<2	12	11	9.6	12	10	8.2
Benzo(a)anthracene	2.4	2.2	1.8	1.2	<2	14	14	7.2	16	8.4	6.1
Chyrsene	2.4	2.1	1.4	1.8	<2	13	14	10	16	11	7.1
Decafluorobiphenyl	2.0	2.8	3.1	<5	<2	13	12	11	12	11	11
Fluoranthene	2.3	2.0	1.8	1.8	<2	13	13	8.5	15	9.5	7.0
Fluorene	2.3	2.1	2.1	1.5	<2	11	10	8.8	11	9.9	7.0
Naphthalene	3.4	2.2	2.4	<5	<2	14	12	11	12	11	8.3
Phenanthrene	2.4	2.2	2.0	1.9	<2	11	11	9.4	12	10	7.5
Pyrene	2.2	2.0	1.9	1.8	<2	12	13	9.1	15	10	7.3
Actual Concentration	2.5	2.5	2.5	2.5	2.5	10	10	10	10	10	10

Footnotes appear on the following page.

TABLE C-8. (Continued)

	C	C	C	C	C	C	C	D	D	D	D	D	D	E	E	E	E	E	E
PESTICIDES/PCBs																			
Arochlor 1254																			
Tetrachlorobiphenyl[a,b]	177	97	102	100	110	160	150	330	360	320	370	340	350	930	970	1000	630	650	560
Pentachlorobiphenyl[a,b]	126	98	97	100	100	170	150	360	400	350	420	370	360	1020	380	870	740	570	530
Hexachlorobiphenyl[a,b]	123	89	114	99	130	160	130	360	400	280	450	310	380	990	800	890	540	500	580
Heptachlor	66	42	46	43	48	41	57	150	130	79	130	80	100	320	290	300	340	250	250
Lindane	51	40	45	44	50	52	49	120	110	110	120	120	110	300	230	260	230	190	220
Toxaphene[a,b]	50	103	130	120	150	160	140	330	350	300	390	330	340	840	590	660	540	570	610
PHENOLS																			
2,4-Dimethylphenol	62	38	45	43	50	44	48	120	120	110	140	120	110	280	240	260	240	210	260
Phenol	63	39	48	41	50	48	56	90	140	99	140	100	100	260	230	240	250	210	230
PHTHALATES																			
Bis(2-ethylhexyl)phthalate	79	31	56	34	60	46	55	140	120	110	130	110	110	390	240	260	270	330	220
Butylbenzylphthalate	88	26	51	26	51	42	54	150	130	100	130	110	100	390	250	250	240	170	270
Diethylphthalate	64	41	48	45	53	52	43	100	110	120	120	130	120	270	220	250	250	240	220
Dimethylphthalate	58	41	46	45	50	51	44	100	110	120	120	140	120	250	230	250	230	220	230
Di-n-butylphthalate	73	41	46	48	53	55	52	120	130	110	150	130	120	270	210	250	220	230	160
Di-n-octylphthalate		37	53	42	60	42	63	160	120	82	130	90	100	470	200	230	250	170	220
PAHs																			
Acenaphthene	53	39	47	43	51	52	45	100	110	130	120	140	120	270	220	240	230	230	220
Anthracene	53	42	45	45	48	51	47	100	120	120	120	130	120	260	220	230	220	240	210
Benzo(a)anthracene	65	42	41	49	47	37	60	180	120	110	140	120	90	470	250	300	250	190	280
Chyrsene	58	42	40	48	46	41	59	140	110	110	120	120	90	370	220	250	230	140	260
Decafluorobiphenyl	49	33	37	34	38	61	43	84	110	140	110	150	130	270	200	210	190	170	160
Fluoranthene	53	43	41	49	46	50	51	120	110	110	130	120	110	300	230	260	210	250	210
Fluorene	54	41	46	46	51	50	45	110	110	130	130	140	120	280	240	270	240	240	230
Naphthalene	64	42	50	45	54	57	51	75	110	120	120	130	120	190	160	180	190	210	170
Phenanthrene	53	41	47	46	51	51	46	110	110	110	120	130	110	250	230	260	240	230	230
Pyrene	53	43	40	48	45	50	51	120	110	110	130	120	110	310	230	260	210	240	210
Actual Concentration	50	50	50	50	50	50	50	100	100	100	100	100	100	250	250	250	250	250	250

(a) Actual Concentrations were A = 7.5 µg/ml, B = 30 µg/ml, C = 150 µg/ml, D = 300 µg/ml, E = 750 µg/ml.
(b) The single most intense isomer was used to calculate the total amount of Arochlor 1254 and toxaphene.

TABLE C-9. SUMMARY OF THE QUANTIFICATION OF PRIORITY POLLUTANT STANDARDS

	A			B			C			D			E			Normalized	
	Mean	RSD	Mean Deviation %	Mean	RSD	Mean Deviation %	Mean	RSD	Mean Deviation %	Mean	RSD	Mean Deviation %	Mean	RSD	Mean Deviation %	Mean	S
Pesticides																	
Arochlor 1254																	
Tetrachlorobiphenyl[a,b]	6.8	22.0	9.3	31	12.2	3.3	130	26.8	13	350	5.4	17	790	24.9	5.3	1.00	.21
Pentachlorobiphenyl[a,b]	6.3	29.3	16	31	23.9	3.3	120	24.6	20	380	7.3	27	690	34.5	8.0	.98	.27
Hexachlorobiphenyl[a,b]	6.3	13.1	16	35	17.7	17	120	19.6	20	360	16.9	20	720	28.5	4.0	1.00	.25
Heptachlor	2.2	40.9	12	9.3	29.5	7.0	49	18.9	2.0	110	26.4	10	290	12.5	16	1.04	.24
Lindane	2.1	24.4	16	10	20.3	0	47	9.3	6.0	120	4.8	20	240	15.8	4.0	.99	.17
Toxaphene[a,b]	5.1	N/A	32	37	20.4	23	120	30.6	20	340	8.7	13	640	17.0	15	.99	.27
Phenols																	
2,4-Dimethylphenol	2.1	26.6	16	11	17.4	10	47	16.1	6.0	120	9.1	20	250	9.7	0	1.02	.18
Phenol	2.9	32.2	16	10	22.1	0	49	16.8	2.0	110	20.1	10	240	7.4	4.0	1.03	.19
Phthalates																	
Bis(2-ethylhexyl) phthalate	2.4	41.5	4.0	11	49.2	10	52	31.8	4.0	120	10.5	20	290	22.3	16	1.09	.34
Butylbenzylphthalate	2.2	50.1	12	11	36.8	10	48	43.6	4.0	120	16.7	20	260	27.4	4.0	1.05	.35
Diethylphthalate	2.1	23.7	16	10	17.7	0	49	15.8	2.0	120	8.9	20	240	8.0	4.0	1.01	.17
Dimethylphthalate	2.2	15.2	12	10	17.5	0	48	11.8	4.0	120	11.2	20	240	5.2	4.0	1.00	.16
Di-n-butylphthalate	1.9	28.8	24	11	27.4	10	53	19.4	6.0	130	10.8	30	220	16.9	12	1.03	.25
Di-n-octylphthalate	2.4	48.5	4.0	13	41.8	30	50	21.6	0	110	25.5	10	260	42.0	4.0	1.03	.37
PAHs																	
Acenaphthene	2.2	13.7	12	10	13.9	0	47	11.0	6.0	120	11.8	20	240	8.0	4.0	1.00	.16
Anthracene	2.4	12.3	4.0	10	14.2	0	47	8.0	6.0	120	10.5	20	230	7.8	8.0	1.01	.14
Benzo(a)anthracene	1.9	27.9	24	11	38.4	10	49	21.2	2.0	130	24.3	30	290	33.0	16	1.07	.35
Chrysene	1.9	22.2	24	12	26.7	20	48	16.5	4.0	120	14.3	20	250	30.4	0	1.02	.26
Decafluorobiphenyl	2.6	21.6	4.0	12	7.0	20	42	23.7	16	120	19.9	20	200	19.5	20	1.00	.25
Fluoranthene	2.0	12.0	20	11	28.3	10	48	9.2	4.0	120	7.0	20	240	14.2	4.0	1.01	.20
Fluorene	2.0	17.3	20	9.6	15.8	4.0	48	9.2	4.0	120	9.8	20	250	8.0	0	1.00	.17
Naphthalene	2.7	24.1	8.0	11	16.4	10	52	14.2	4.0	110	17.3	10	180	9.6	28	1.02	.22
Phenanthrene	2.1	10.4	16	10	15.6	0	48	9.0	4.0	120	7.3	20	240	5.3	4.0	1.00	.13
Pyrene	2.0	8.6	20	11	25.3	10	47	9.9	6.0	120	7.0	20	240	15.5	4.0	1.01	.19
Actual Concentration	2.5			10			50			100			250				

(a) Actual Concentrations were A = 7.5 µg/ml, B = 30 µg/ml, C = 150 µg/ml, D = 300 µg/ml, E = 750 µg/ml.
(b) The single most intense isomer was used to calculate the total amount of Arochlor 1254 and toxaphene.